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     (FILE 'HOME' ENTERED AT 12:41:45 ON 22 MAR 2006)

     FILE 'CAPLUS' ENTERED AT 12:41:55 ON 22 MAR 2006
L1                STRUCTURE UPLOADED
                  S L1

     FILE 'REGISTRY' ENTERED AT 12:42:17 ON 22 MAR 2006
L2             0 S L1

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L3             0 S L2
                 S L1

     FILE 'REGISTRY' ENTERED AT 12:42:26 ON 22 MAR 2006
L4             1 S L1 FULL

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L5             2 S L4 FULL

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L6             2 S 117528-59-3/PREP
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L9                STRUCTURE UPLOADED
                  S L9

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     FILE 'CAPLUS' ENTERED AT 13:42:49 ON 22 MAR 2006
L11            0 S L10
                 S L9

     FILE 'REGISTRY' ENTERED AT 13:42:56 ON 22 MAR 2006
L12            1 S L9 FULL

     FILE 'CAPLUS' ENTERED AT 13:42:56 ON 22 MAR 2006
L13            3 S L12 FULL

     FILE 'STNGUIDE' ENTERED AT 13:45:26 ON 22 MAR 2006

     FILE 'CAPLUS' ENTERED AT 13:51:46 ON 22 MAR 2006
L14               STRUCTURE UPLOADED
                  S L14

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L15            0 S L14

     FILE 'CAPLUS' ENTERED AT 13:52:10 ON 22 MAR 2006
L16            0 S L15
                 S L14

     FILE 'REGISTRY' ENTERED AT 13:52:15 ON 22 MAR 2006
L17            1 S L14 FULL

     FILE 'CAPLUS' ENTERED AT 13:52:16 ON 22 MAR 2006
L18            4 S L17 FULL
L19            4 S 195532-66-2/PREP
L20            0 S 195532-66-2/PUR
L21            0 S 195532-66-2/PROC
L22            0 S L19 AND PY<1997

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DOCUMENT NUMBER: 129:316044
TITLE: 3-Cyano-2,4,5-trifluorobenzoyl fluoride and intermediates for its production
INVENTOR(S): Marhold, Albrecht; Wolfrum, Peter
PATENT ASSIGNEE(S): Bayer Aktiengesellschaft, Germany
SOURCE: PCT Int. Appl., 30 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9847862	A1	19981029	WO 1998-EP2175	19980414
W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
DE 19717231	A1	19981029	DE 1997-19717231	19970424
CA 2287176	AA	19981029	CA 1998-2287176	19980414
AU 9872163	A1	19981113	AU 1998-72163	19980414
EP 977729	A1	20000209	EP 1998-919266	19980414
EP 977729	B1	20020313		
R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, IE				
JP 2001521534	T2	20011106	JP 1998-544950	19980414
AT 214365	E	20020315	AT 1998-919266	19980414
ES 2174431	T3	20021101	ES 1998-919266	19980414
CN 1119324	B	20030827	CN 1998-804339	19980414
IL 131974	A1	20040219	IL 1998-131974	19980414
US 6229040	B1	20010508	US 1999-403263	19991015
HK 1027555	A1	20040514	HK 2000-106792	20001025
US 2001023300	A1	20010920	US 2001-814132	20010321
US 6541675	B2	20030401		
US 2003092929	A1	20030515	US 2002-277310	20021022
US 6706918	B2	20040316		
CN 1436771	A	20030820	CN 2002-148153	20021031
US 2004167350	A1	20040826	US 2003-749593	20031231

PRIORITY APPLN. INFO.:

DE 1997-19717231	A	19970424
WO 1998-EP2175	W	19980414
US 1999-403263	A3	19991015
US 2001-814132	A1	20010321
US 2002-227310	A3	20020826

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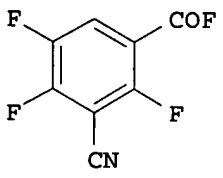


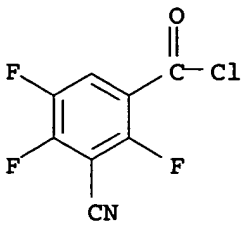
I

AB 3-Cyano-2,4,5-trifluorobenzoyl fluoride (I) is prepared starting from 5-fluoro-m-xylene and proceeding via 2,4-dichloro-5-fluoro-1,3-dimethylbenzene, 2,4-dichloro-5-fluoro-3-(dichloromethyl)-1-(trichloromethyl)benzene, 2,4-dichloro-5-fluoro-3-(dichloromethyl)benzoic acid, 2,4-dichloro-5-fluoro-3-formylbenzoic acid (II), the oxime of II, and 2,4-dichloro-3-cyano-5-fluorobenzoyl chloride.

IT **195532-66-2P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

RN 195532-66-2 CAPLUS

CN Benzoyl chloride, 3-cyano-2,4,5-trifluoro- (9CI) (CA INDEX NAME)



REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L18 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1998:406218 CAPLUS
DOCUMENT NUMBER: 129:81719
TITLE: Preparation of 7-(2-oxa-5,8-diazabicyclo[4.3.0]non-8-yl)quinolone- and -naphthyridonecarboxylic acid derivatives for therapy of Helicobacter pylori infections and associated gastroduodenal illnesses
INVENTOR(S): Matzke, Michael; Petersen, Uwe; Jaetsch, Thomas; Bartel, Stephan; Schenke, Thomas; Himmler, Thomas; Baasner, Bernd; Werling, Hans-Otto; Schaller, Klaus; Labischinski, Harald
PATENT ASSIGNEE(S): Bayer A.-G., Germany
SOURCE: Ger. Offen., 16 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 19652239	A1	19980618	DE 1996-19652239	19961216
CA 2274894	AA	19980625	CA 1997-2274894	19971204
WO 9826779	A1	19980625	WO 1997-EP6781	19971204
W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW				
RW: GH, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
AU 9858541	A1	19980715	AU 1998-58541	19971204
AU 717751	B2	20000330		
EP 946176	A1	19991006	EP 1997-954354	19971204
EP 946176	B1	20020327		
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BR 9714032	A	20000509	BR 1997-14032	19971204
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JP 3463939	B2	20031105		
JP 2000351781	A2	20001219	JP 2000-154543	19971204
NZ 336228	A	20001222	NZ 1997-336228	19971204
AT 214929	E	20020415	AT 1997-954354	19971204
PT 946176	T	20020830	PT 1997-954354	19971204
ES 2175519	T3	20021116	ES 1997-954354	19971204
SK 283224	B6	20030304	SK 2000-1496	19971204
SK 283223	B6	20030304	SK 1999-795	19971204
EE 4090	B1	20030815	EE 1999-248	19971204
IL 130311	A1	20040104	IL 1997-130311	19971204
BG 64615	B1	20050930	BG 1999-103474	19990608
NO 9902903	A	19990614	NO 1999-2903	19990614

US 6133260	A	20001017	US 1999-319888		19990614
US 6432948	B1	20020813	US 1999-436316		19991108
NZ 506162	A	20010629	NZ 2000-506162		20000804
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			JP 1998-527250	A3	19971204
			NZ 1997-336228	A	19971204
			WO 1997-EP6781	W	19971204
			US 1999-319888	A3	19990614

OTHER SOURCE(S): MARPAT 129:81719

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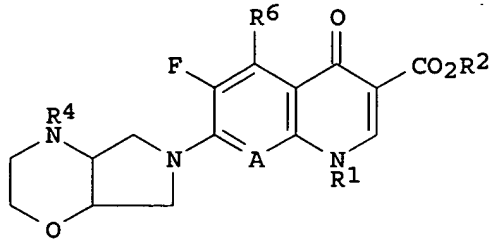

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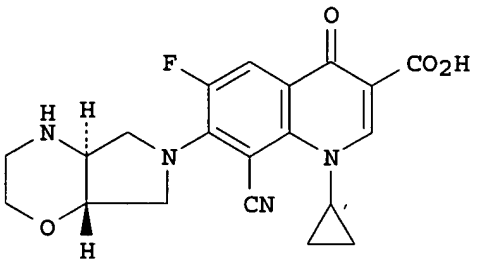


II

AB The title compds. [I; A = N, CR3; R1 = C1-4 (halo)alkyl; (fluoro)phenyl; (fluoro)cyclopropyl; R2 = H, (un)substituted C1-4 alkyl, (5-methyl-2-oxo-1,3-dioxol-4-yl)methyl; R3 = H, halo, Me, MeO, CHF2O, cyano; R1R3 = OCH2CHMe or OCH2NMe bound to A via O atom; R4 = H, PhCH2, C1-3 alkyl, (5-methyl-2-oxo-1,3-dioxol-4-yl)methyl, CH:CHCO2R5, etc.; R5 = Me, Et; R6 = H, amino, OH, Me, halo], their racemates, diastereomer mixts., enantiomers, diastereoisomers and pharmaceutically acceptable hydrates and salts, were prepared For example, stirring 200 mg Et 8-cyano-1-cyclopropyl-6,7-difluoro-1,4-dihydro-4-oxo-3-quinolinecarboxylate (7-step preparation from 3-bromo-2,4,5-trifluorobenzoyl fluoride given) with 97 mg (1S,6S)-2-oxa-5,8-diazabicyclo[4.3.0]nonane and 0.9 mL Et3N for 25 h at 40-45° in 30 mL MeCN under Ar, working up the product, saponifying by stirring with 30 mg LiOH·H2O in aqueous THF for 16 h at ambient temperature and acidifying the carboxylate salt with HCl gave 57% II-HCl (m. >300°). II at 2 + 10 mg/kg in H. pylori-infected mice gave after 7 days 100% clearing, vs. 0% for ciprofloxacin.

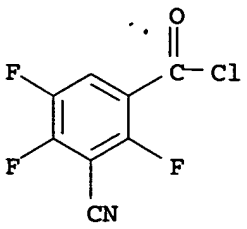
IT **195532-66-2P**

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation and condensation with Et 3-dimethylaminoacrylate; preparation of (oxadiazabicyclononyl)quinolone- and -naphthyridonecarboxylic acid derivs. as drugs for therapy of Helicobacter pylori infections and associated gastroduodenal illnesses)

RN 195532-66-2 CAPLUS

CN Benzoyl chloride, 3-cyano-2,4,5-trifluoro- (9CI) (CA INDEX NAME)



L18 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1998:406214 CAPLUS
DOCUMENT NUMBER: 129:81718
TITLE: Use of 7-(1-aminomethyl-2-oxa-7-azabicyclo(3.3.0)oct-7-yl)-quinolonecarboxylates, -naphthyridonecarboxylates, and related compounds for Helicobacter pylori infection therapy and associated gastroduodenal illnesses.
INVENTOR(S): Petersen, Uwe; Matzke, Michael; Jaetsch, Thomas; Schenke, Thomas; Himmler, Thomas; Bartel, Stephan; Baasner, Bernd; Werling, Hans-Otto; Schaller, Klaus; Labischinski, Harald; Endermann, Rainer
PATENT ASSIGNEE(S): Bayer A.-G., Germany
SOURCE: Ger. Offen., 32 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 19652219	A1	19980618	DE 1996-19652219	19961216
CA 2274892	AA	19980625	CA 1997-2274892	19971203
WO 9826768	A2	19980625	WO 1997-EP6751	19971203
WO 9826768	A3	19980806		
W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
AU 9858538	A1	19980715	AU 1998-58538	19971203
EP 944385	A2	19990929	EP 1997-954350	19971203
EP 944385	B1	20021016		
R: DE, ES, FR, GB, IT				
JP 2001506242	T2	20010515	JP 1998-527239	19971203
ES 2184150	T3	20030401	ES 1997-954350	19971203
US 6288081	B1	20010911	US 1999-319848	19990806
US 2001036941	A1	20011101	US 2001-829776	20010410

PRIORITY APPLN. INFO.:
DE 1996-19652219 A 19961216
WO 1997-EP6751 W 19971203
US 1999-319848 A3 19990806

OTHER SOURCE(S): MARPAT 129:81718
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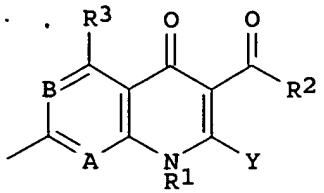
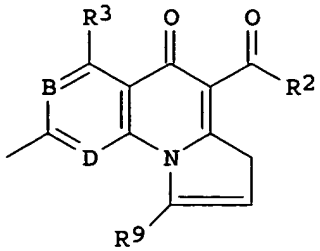
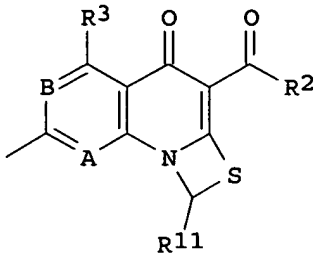
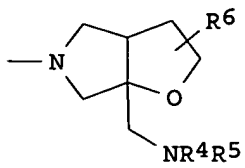
Q1 = 

Q2 = 

Q3 = 

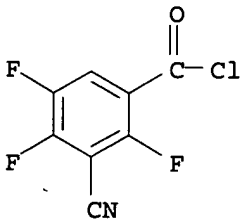
Q4 = 

AB Use of TQ [Q = Q1-Q3; T = Q4; A = N, CR7; B = N, CH, CF, CCl, CNO2, CNH2; D = N, CR10; Y = H; YR2 = SNH; R1 = (substituted) alkyl, alkenyl, cycloalkyl, bicyclo[1.1.1]pent-1-yl, 1,1-dimethylpropargyl, 3-oxetanyl, MeO, amino, etc.; R2 = OH, (substituted) alkoxy, PhCH2O, allyloxy, propargyloxy, acetoxymethoxy, etc.; R3 = H, amino, OH, Me, halo; R7 = H, halo, CF3, OMe, OCHF2, Me, cyano, CH:CH2, C.tplbond.CH; R1R7 = CH2CHMe, SCH2CH2, SCH2CHMe, etc.; R9 = H, (substituted) alkyl; R10 = H, halo, CF3, OMe, OCHF2, Me; R9R10 = OCH2, NHCH2, NMeCH2, SCH2, etc.; R11 = H, Me, CH2F; R4 = H, Me, Et, (substituted) acyl, alkoxycarbonyl, aminocarbonyl, etc.; R5 = H, Me, Et; R6 = H, Me], for treatment of Helicobacter pylori infection and associated gastroduodenal illnesses is claimed. Thus, 7-(1-aminomethyl-2-oxa-7-azobicyclo[3.3.0]oct-7-yl)-1-cyclopropyl-8-difluoromethoxy-6-fluoro-1,4-dihydro-4-oxo-3-quinolinecarboxylic acid (preparation given) had a min. inhibitory concentration of 0.006 mg/L against H. pylori 008.

IT **195532-66-2P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
((aminomethyloxaazabicyclooctyl)quinolonecarboxylates, -naphthyridonecarboxylates, and related compds. for Helicobacter pylori infection therapy and associated gastroduodenal illnesses)

RN 195532-66-2 CAPLUS

CN Benzoyl chloride, 3-cyano-2,4,5-trifluoro- (9CI) (CA INDEX NAME)



L18 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1997:579724 CAPLUS

DOCUMENT NUMBER: 127:248093

TITLE: 8-Cyano-1-cyclopropyl-7-(2,8-diazabicyclo[4.3.0]nonan-8-yl)-6-fluoro-1,4-dihydro-4-oxo-3-quinolinecarboxylic acid derivatives

INVENTOR(S): Bartel, Stefan; Jaetsch, Thomas; Himmler, Thomas; Rast, Hans-Georg; Hallenbach, Werner; Heinen, Ernst; Pirro, Franz; Scheer, Martin; Stegemann, Michael; Stupp, Hans-Peter; Wetzstein, Heinz-Georg

PATENT ASSIGNEE(S): Bayer A.-G., Germany; Bartel, Stefan; Jaetsch, Thomas;

Himmler, Thomas; Rast, Hans-Georg; Hallenbach, Werner;
Heinen, Ernst; Pirro, Franz; Scheer, Martin; et al.
SOURCE: PCT Int. Appl., 36 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9731001	A1	19970828	WO 1997-EP637	19970212
W: AU, BB, BG, BR, BY, CA, CN, CZ, HU, IL, JP, KR, KZ, LK, MX, NO, NZ, PL, RO, RU, SK, TR, UA, US				
RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
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ZA 9701507	A	19970916	ZA 1997-1507	19970202
CA 2247020	AA	19970828	CA 1997-2247020	19970212
CA 2247020	C	20051108		
AU 9717689	A1	19970910	AU 1997-17689	19970212
AU 715341	B2	20000120		
EP 882049	A1	19981209	EP 1997-903260	19970212
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CN 1073112	B	20011017		
BR 9707606	A	19990727	BR 1997-7606	19970212
NZ 331468	A	20000228	NZ 1997-331468	19970212
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PL 186737	B1	20040227	PL 1997-328577	19970212
SK 284542	B6	20050602	SK 1998-1146	19970212
TW 390879	B	20000521	TW 1997-86101994	19970220
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PRIORITY APPLN. INFO.:

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OTHER SOURCE(S): MARPAT 127:248093
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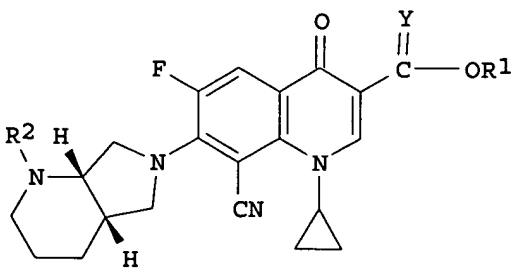


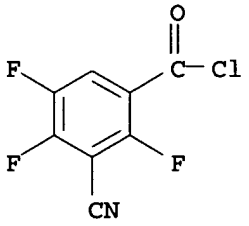
I

AB Title compds. I [R1 = H, alkyl, optionally substituted by OH, OMe, NH2, NHMe, NMe2, or (5-methyl-2-oxo-1,3-dioxol-4-yl)methyl; R2 = H, benzyl, alkyl, (5-methyl-2-oxo-1,3-dioxol-4-yl)methyl, CH=CHCO2R3, CH2CH2CO2R3, CH2CH2CN, CH2CH2COMe, CH2COMe; R3 = Me, Et, R4(NHCHR5CO)n; R4 = H, alkyl, CO2CMe3; R5 = H, alkyl, hydroxyalkyl, aminoalkyl, thioalkyl, carboxyalkyl, benzyl; n = 1, 2; Y = O, S] were prepared for use as antibacterial agents. Thus, I [R1 = OH, R2 = H, Y = O] was prepared by aminating the 7-chloroquinoline. I [R1 = OH, R2 = H, Y = O] had min. inhibitory concns. against a number of bacteria that were superior to those of enrofloxacin.

IT **195532-66-2P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation of diazabicylononylquinolinecarboxylic acid derivs. as bactericides)

RN 195532-66-2 CAPLUS

CN Benzoyl chloride, 3-cyano-2,4,5-trifluoro- (9CI) (CA INDEX NAME)



L25 STRUCTURE UPLOADED

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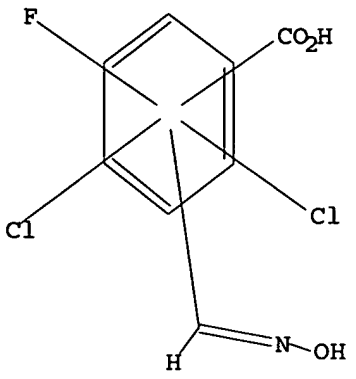
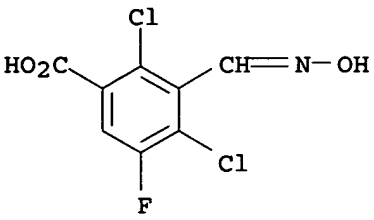
L25 HAS NO ANSWERS
L25 STR



Structure attributes must be viewed using STN Express query preparation.

=>

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and chlorination-dehydration of)
RN 214774-57-9 CAPLUS
CN Benzoic acid, 2,4-dichloro-5-fluoro-3-[(hydroxyimino)methyl]- (9CI) (CA INDEX NAME)



REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

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L25 STRUCTURE UPLOADED

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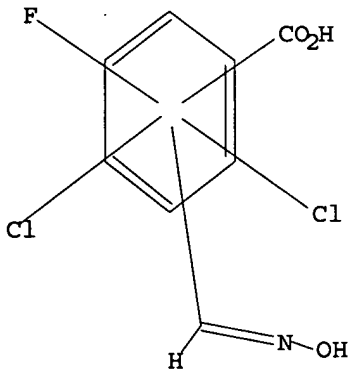
L25 HAS NO ANSWERS
L25 STR



Structure attributes must be viewed using STN Express query preparation.

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L24 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1998:709043 CAPLUS
DOCUMENT NUMBER: 129:316044
TITLE: 3-Cyano-2,4,5-trifluorobenzoyl fluoride and intermediates for its production
INVENTOR(S): Marhold, Albrecht; Wolfrum, Peter
PATENT ASSIGNEE(S): Bayer Aktiengesellschaft, Germany
SOURCE: PCT Int. Appl., 30 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9847862	A1	19981029	WO 1998-EP2175	19980414
W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
DE 19717231	A1	19981029	DE 1997-19717231	19970424
CA 2287176	AA	19981029	CA 1998-2287176	19980414
AU 9872163	A1	19981113	AU 1998-72163	19980414
EP 977729	A1	20000209	EP 1998-919266	19980414
EP 977729	B1	20020313		
R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, IE				
JP 2001521534	T2	20011106	JP 1998-544950	19980414
AT 214365	E	20020315	AT 1998-919266	19980414
ES 2174431	T3	20021101	ES 1998-919266	19980414
CN 1119324	B	20030827	CN 1998-804339	19980414
IL 131974	A1	20040219	IL 1998-131974	19980414
US 6229040	B1	20010508	US 1999-403263	19991015
HK 1027555	A1	20040514	HK 2000-106792	20001025
US 2001023300	A1	20010920	US 2001-814132	20010321
US 6541675	B2	20030401		
US 2003092929	A1	20030515	US 2002-277310	20021022
US 6706918	B2	20040316		
CN 1436771	A	20030820	CN 2002-148153	20021031
US 2004167350	A1	20040826	US 2003-749593	20031231

PRIORITY APPLN. INFO.:

DE 1997-19717231	A	19970424
WO 1998-EP2175	W	19980414
US 1999-403263	A3	19991015
US 2001-814132	A1	20010321
US 2002-227310	A3	20020826

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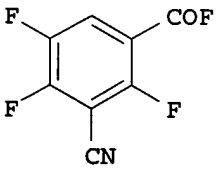

I

AB 3-Cyano-2,4,5-trifluorobenzoyl fluoride (I) is prepared starting from 5-fluoro-m-xylene and proceeding via 2,4-dichloro-5-fluoro-1,3-dimethylbenzene, 2,4-dichloro-5-fluoro-3-(dichloromethyl)-1-(trichloromethyl)benzene, 2,4-dichloro-5-fluoro-3-(dichloromethyl)benzoic acid, 2,4-dichloro-5-fluoro-3-formylbenzoic acid (II), the oxime of II, and 2,4-dichloro-3-cyano-5-fluorobenzoyl chloride.

IT **214774-57-9P**

L1 . STRUCTURE UPLOADED

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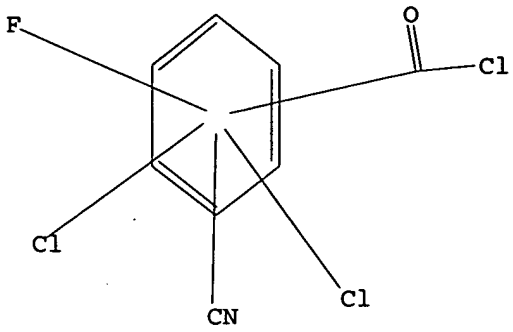
L1 HAS NO ANSWERS
L1 STR



Structure attributes must be viewed using STN Express query preparation.

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REGISTRY INITIATED
Substance data SEARCH and crossover from CAS REGISTRY in progress...
Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

SAMPLE SEARCH INITIATED 12:42:18 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED - 5 TO ITERATE

100.0% PROCESSED 5 ITERATIONS 0 ANSWERS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED ITERATIONS: 5 TO 234
PROJECTED ANSWERS: 0 TO 0

L2 0 SEA SSS SAM L1

L3 0 L2

=> s l1 full
REGISTRY INITIATED
Substance data SEARCH and crossover from CAS REGISTRY in progress...
Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

FULL SEARCH INITIATED 12:42:27 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 138 TO ITERATE

100.0% PROCESSED 138 ITERATIONS 1 ANSWERS
SEARCH TIME: 00.00.01

L4 1 SEA SSS FUL L1

L5 2 L4

=> d 1-2 ibib abs hitstr

L5 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1998:709043 CAPLUS
DOCUMENT NUMBER: 129:316044
TITLE: 3-Cyano-2,4,5-trifluorobenzoyl fluoride and intermediates for its production
INVENTOR(S): Marhold, Albrecht; Wolfrum, Peter
PATENT ASSIGNEE(S): Bayer Aktiengesellschaft, Germany
SOURCE: PCT Int. Appl., 30 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9847862	A1	19981029	WO 1998-EP2175	19980414
W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
DE 19717231	A1	19981029	DE 1997-19717231	19970424
CA 2287176	AA	19981029	CA 1998-2287176	19980414
AU 9872163	A1	19981113	AU 1998-72163	19980414
EP 977729	A1	20000209	EP 1998-919266	19980414
EP 977729	B1	20020313		
R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, IE				
JP 2001521534	T2	20011106	JP 1998-544950	19980414
AT 214365	E	20020315	AT 1998-919266	19980414
ES 2174431	T3	20021101	ES 1998-919266	19980414
CN 1119324	B	20030827	CN 1998-804339	19980414
IL 131974	A1	20040219	IL 1998-131974	19980414
US 6229040	B1	20010508	US 1999-403263	19991015
HK 1027555	A1	20040514	HK 2000-106792	20001025
US 2001023300	A1	20010920	US 2001-814132	20010321
US 6541675	B2	20030401		
US 2003092929	A1	20030515	US 2002-277310	20021022
US 6706918	B2	20040316		
CN 1436771	A	20030820	CN 2002-148153	20021031
US 2004167350	A1	20040826	US 2003-749593	20031231

PRIORITY APPLN. INFO.:

DE 1997-19717231	A	19970424
WO 1998-EP2175	W	19980414
US 1999-403263	A3	19991015
US 2001-814132	A1	20010321
US 2002-227310	A3	20020826

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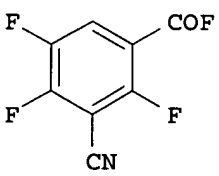


I

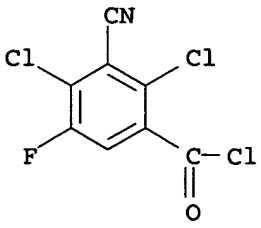
AB 3-Cyano-2,4,5-trifluorobenzoyl fluoride (I) is prepared starting from 5-fluoro-m-xylene and proceeding via 2,4-dichloro-5-fluoro-1,3-dimethylbenzene, 2,4-dichloro-5-fluoro-3-(dichloromethyl)-1-(trichloromethyl)benzene, 2,4-dichloro-5-fluoro-3-(dichloromethyl)benzoic

acid, 2,4-dichloro-5-fluoro-3-formylbenzoic acid (II), the oxime of II, and 2,4-dichloro-3-cyano-5-fluorobenzoyl chloride.

IT 117528-59-3P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and fluorination of)

RN 117528-59-3 CAPLUS

CN Benzoyl chloride, 2,4-dichloro-3-cyano-5-fluoro- (9CI) (CA INDEX NAME)



REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 2 OF 2 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1988:630824 CAPLUS

DOCUMENT NUMBER: 109:230824

TITLE: 8-Cyano-1-cyclopropylquinolonecarboxylic acids as antibacterial agents

INVENTOR(S): Schriewer, Michael; Grohe, Klaus; Petersen, Uwe; Haller, Ingo; Metzger, Karl Georg; Endermann, Rainer; Zeiler, Hans Joachim

PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.

SOURCE: Ger. Offen., 20 pp.
CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3702393	A1	19880811	DE 1987-3702393	19870128
US 4908366	A	19900313	US 1988-144884	19880114
EP 276700	A1	19880803	EP 1988-100503	19880115
R: AT, BE, CH, DE, ES, FR, GB, GR, IT, LI, NL, SE				
CA 1314544	A1	19930316	CA 1988-557311	19880126
JP 63201170	A2	19880819	JP 1988-14771	19880127
US 5051418	A	19910924	US 1989-434666	19891113
US 5190955	A	19930302	US 1991-645751	19910125

PRIORITY APPLN. INFO.:
DE 1987-3702393 A 19870128
US 1988-144884 A3 19880114
US 1989-434666 A3 19891113

OTHER SOURCE(S): CASREACT 109:230824; MARPAT 109:230824

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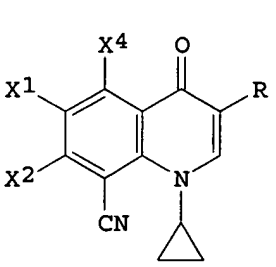
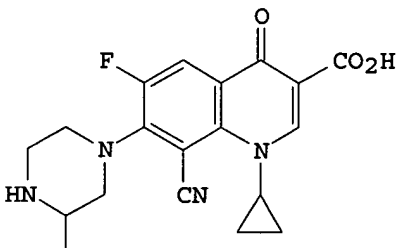


I



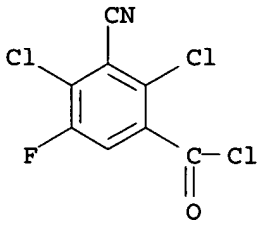
III

AB The title compds. [I; R = CO2H, cyano, CO2R1, CONR2R3; R1 = alkyl; R2 = H, alkyl; R3 = R2, (un)substituted Ph; X1 = H, NO2, alkyl, halo; X2 = heterocyclyl; X4 = H, halo, alkyl] were prepared as antibacterial agents (no data). 2,4,5,3-Cl2F(NC)C6HCOCH2CO2Et (preparation given) was heated 2 h at 150° with HC(OEt)3 in Ac2O to give 2,4,5,3-Cl2F(NC)C6HCOC(:CHR4)CO2Et (II; R4 = OEt) which was stirred 2 h with cyclopropylamine in EtOH to give II (R = cyclopropylamino). The latter was stirred 24 h in dioxane containing KOCMe3 to give, after saponification, I (R = CO2H, X1 = F, X2 = Cl, X4 = H) which was heated 3 h in dioxane with 2-methylpiperazine to give title compound III. Tablets were prepared each containing III 583.0, cellulose 55.0, starch 72.0, polyvinylpyrrolidone 30.0, silica 5.0, and Mg stearate 5.0 mg coated with poly(O-hydroxypropyl-O-methyl)cellulose 6.0, Macrogol 4000 2.0, TiO2 2.0 mg, and polyethyleneglycol (no amount given).

IT **117528-59-3P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reaction of, in preparation of antibacterial agents)

RN 117528-59-3 CAPLUS

CN Benzoyl chloride, 2,4-dichloro-3-cyano-5-fluoro- (9CI) (CA INDEX NAME)



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L9 STRUCTURE UPLOADED

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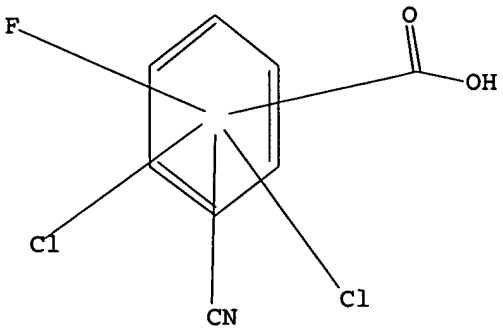
L9 HAS NO ANSWERS
L9 STR



Structure attributes must be viewed using STN Express query preparation.

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REGISTRY INITIATED
Substance data SEARCH and crossover from CAS REGISTRY in progress....
Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

SAMPLE SEARCH INITIATED 13:42:49 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED - 20 TO ITERATE

100.0% PROCESSED 20 ITERATIONS 0 ANSWERS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED ITERATIONS: 132 TO 668
PROJECTED ANSWERS: 0 TO 0

L10 0 SEA SSS SAM L9

L11 0 L10

=> s l9 full
REGISTRY INITIATED
Substance data SEARCH and crossover from CAS REGISTRY in progress...
Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

FULL SEARCH INITIATED 13:42:56 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 260 TO ITERATE

100.0% PROCESSED 260 ITERATIONS 1 ANSWERS
SEARCH TIME: 00.00.01

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L13 3 L12

=> d 1-3 ibib abs hitstr

L13 ANSWER 1 OF 3 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1999:101296 CAPLUS
DOCUMENT NUMBER: 130:139168
TITLE: Preparation of 3-cyano-2,4-dihalo-5-fluorobenzoic acid by hydrolysis of the corresponding amides, nitriles, or esters.
INVENTOR(S): Hallenbach, Werner; Marhold, Albrecht
PATENT ASSIGNEE(S): Bayer A.-G., Germany
SOURCE: Ger. Offen., 18 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 19733243	A1	19990204	DE 1997-19733243	19970801
CA 2298805	AA	19990211	CA 1998-2298805	19980718
WO 9906360	A1	19990211	WO 1998-EP4468	19980718
W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
AU 9891544	A1	19990222	AU 1998-91544	19980718
AU 744367	B2	20020221		
EP 1001929	A1	20000524	EP 1998-943740	19980718
EP 1001929	B1	20030326		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, NL, SE, PT, IE, FI				
BR 9811579	A	20000822	BR 1998-11579	19980718
JP 2001512098	T2	20010821	JP 2000-505122	19980718
NZ 502587	A	20020531	NZ 1998-502587	19980718
AT 235461	E	20030415	AT 1998-943740	19980718
PT 1001929	T	20030731	PT 1998-943740	19980718
ES 2190602	T3	20030801	ES 1998-943740	19980718
CN 1125042	B	20031022	CN 1998-807849	19980718
RU 2214998	C2	20031027	RU 2000-105245	19980718
US 6462218	B1	20021008	US 2000-463272	20000124
MX 200000861	A	20001109	MX 2000-861	20000125
HK 1030598	A1	20040702	HK 2001-101537	20010302

PRIORITY APPLN. INFO.: DE 1997-19733243 A 19970801
WO 1998-EP4468 W 19980718

OTHER SOURCE(S): CASREACT 130:139168
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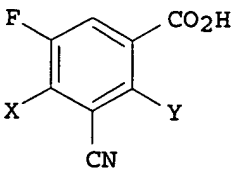
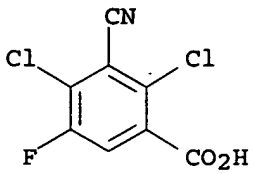


I

AB Title compds. (I; X, Y = halo) were prepared by hydrolysis of the corresponding 3-cyano-2,4-dihalo-5-fluorobenzamides, 1,3-dicyano-2,4-dihalo-5-fluorobenzenes, or 3-cyano-2,4-dihalo-5-fluorobenzoate esters. Thus, 3-cyano-2,4-dichloro-5-fluorobenzamide was refluxed 3 h with concentrate aqueous HCl to give 3-cyano-2,4-dichloro-5-fluorobenzoic acid.
IT **117528-58-2P**

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(preparation of 3-cyano-2,4-dihalo-5-fluorobenzoic acid by hydrolysis of the corresponding amides, nitriles, or esters)
RN 117528-58-2 CAPLUS
CN Benzoic acid, 2,4-dichloro-3-cyano-5-fluoro- (9CI) (CA INDEX NAME)



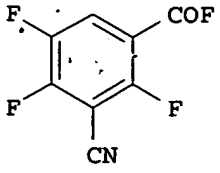
L13 ANSWER 2 OF 3 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1998:709043 CAPLUS
DOCUMENT NUMBER: 129:316044
TITLE: 3-Cyano-2,4,5-trifluorobenzoyl fluoride and intermediates for its production
INVENTOR(S): Marhold, Albrecht; Wolfrum, Peter
PATENT ASSIGNEE(S): Bayer Aktiengesellschaft, Germany
SOURCE: PCT Int. Appl., 30 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9847862	A1	19981029	WO 1998-EP2175	19980414
W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
DE 19717231	A1	19981029	DE 1997-19717231	19970424
CA 2287176	AA	19981029	CA 1998-2287176	19980414
AU 9872163	A1	19981113	AU 1998-72163	19980414
EP 977729	A1	20000209	EP 1998-919266	19980414
EP 977729	B1	20020313		
R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, IE				
JP 2001521534	T2	20011106	JP 1998-544950	19980414
AT 214365	E	20020315	AT 1998-919266	19980414
ES 2174431	T3	20021101	ES 1998-919266	19980414
CN 1119324	B	20030827	CN 1998-804339	19980414
IL 131974	A1	20040219	IL 1998-131974	19980414
US 6229040	B1	20010508	US 1999-403263	19991015
HK 1027555	A1	20040514	HK 2000-106792	20001025
US 2001023300	A1	20010920	US 2001-814132	20010321
US 6541675	B2	20030401		
US 2003092929	A1	20030515	US 2002-277310	20021022
US 6706918	B2	20040316		
CN 1436771	A	20030820	CN 2002-148153	20021031
US 2004167350	A1	20040826	US 2003-749593	20031231

PRIORITY APPLN. INFO.:

DE 1997-19717231	A	19970424
WO 1998-EP2175	W	19980414
US 1999-403263	A3	19991015
US 2001-814132	A1	20010321
US 2002-227310	A3	20020826

GI



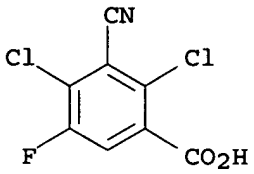
I

AB 3-Cyano-2,4,5-trifluorobenzoyl fluoride (I) is prepared starting from 5-fluoro-m-xylene and proceeding via 2,4-dichloro-5-fluoro-1,3-dimethylbenzene, 2,4-dichloro-5-fluoro-3-(dichloromethyl)-1-(trichloromethyl)benzene, 2,4-dichloro-5-fluoro-3-(dichloromethyl)benzoic acid, 2,4-dichloro-5-fluoro-3-formylbenzoic acid (II), the oxime of II, and 2,4-dichloro-3-cyano-5-fluorobenzoyl chloride.

IT **117528-58-2P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

RN 117528-58-2 CAPLUS

CN Benzoic acid, 2,4-dichloro-3-cyano-5-fluoro- (9CI) (CA INDEX NAME)



REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 3 OF 3 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1988:630824 CAPLUS

DOCUMENT NUMBER: 109:230824

TITLE: 8-Cyano-1-cyclopropylquinolonecarboxylic acids as antibacterial agents

INVENTOR(S): Schriewer, Michael; Grohe, Klaus; Petersen, Uwe; Haller, Ingo; Metzger, Karl Georg; Endermann, Rainer; Zeiler, Hans Joachim

PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.

SOURCE: Ger. Offen., 20 pp.
CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

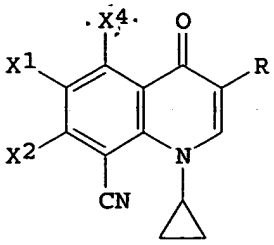
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3702393	A1	19880811	DE 1987-3702393	19870128
US 4908366	A	19900313	US 1988-144884	19880114
EP 276700	A1	19880803	EP 1988-100503	19880115
R: AT, BE, CH, DE, ES, FR, GB, GR, IT, LI, NL, SE				
CA 1314544	A1	19930316	CA 1988-557311	19880126
JP 63201170	A2	19880819	JP 1988-14771	19880127
US 5051418	A	19910924	US 1989-434666	19891113
US 5190955	A	19930302	US 1991-645751	19910125

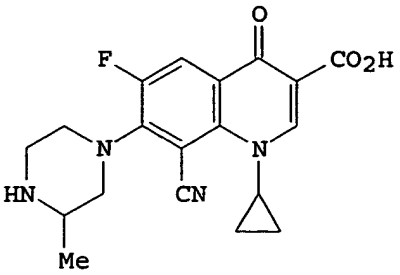
PRIORITY APPLN. INFO.: DE 1987-3702393 A 19870128
US 1988-144884 A3 19880114
US 1989-434666 A3 19891113

OTHER SOURCE(S): CASREACT 109:230824; MARPAT 109:230824

GI



I



III

AB The title compds. [I; R = CO2H, cyano, CO2R1, CONR2R3; R1 = alkyl; R2 = H, alkyl; R3 = R2, (un)substituted Ph; X1 = H, NO2, alkyl, halo; X2 = heterocyclyl; X4 = H, halo, alkyl] were prepared as antibacterial agents (no data). 2,4,5,3-Cl2F(NC)C6HCOCH2CO2Et (preparation given) was heated 2 h at 150° with HC(OEt)3 in Ac2O to give 2,4,5,3-Cl2F(NC)C6HCOC(:CHR4)CO2Et (II; R4 = OEt) which was stirred 2 h with cyclopropylamine in EtOH to give II (R = cyclopropylamino). The latter was stirred 24 h in dioxane containing KOCMe3 to give, after saponification, I (R = CO2H, X1 = F, X2 = Cl, X4 = H) which was heated 3 h in dioxane with 2-methylpiperazine to give title compound III. Tablets were prepared each containing III 583.0, cellulose 55.0, starch 72.0, polyvinylpyrrolidone 30.0, silica 5.0, and Mg stearate 5.0 mg coated with poly(O-hydroxypropyl-O-methyl)cellulose 6.0, Macrogol 4000 2.0, TiO2 2.0 mg, and polyethyleneglycol (no amount given).

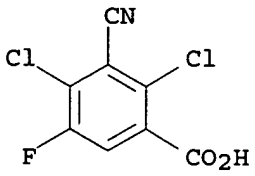
IT **117528-58-2P**

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

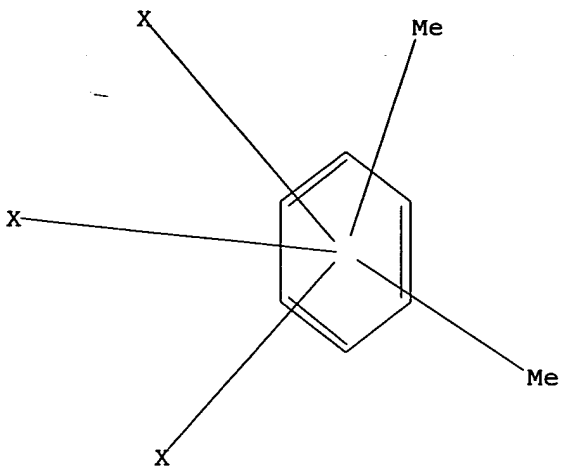
(preparation and reaction of, in preparation of antibacterial agents)

RN 117528-58-2 CAPLUS

CN Benzoic acid, 2,4-dichloro-3-cyano-5-fluoro- (9CI) (CA INDEX NAME)



L8 STR



Structure attributes must be viewed using STN Express query preparation.

=> s l8 sss full
REGISTRY INITIATED
Substance data SEARCH and crossover from CAS REGISTRY in progress...
Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

FULL SEARCH INITIATED 16:21:30 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 1832446 TO ITERATE

42.4% PROCESSED 776666 ITERATIONS 101 ANSWERS

54.6% PROCESSED 1000000 ITERATIONS 109 ANSWERS
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
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FULL FILE PROJECTIONS: ONLINE **INCOMPLETE**
BATCH **INCOMPLETE**
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PROJECTED ANSWERS: 157 TO 241

L9 109 SEA SSS FUL L8

L10 37 L9

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17513355 PY<1997
L11 28 L10 AND PY<1997

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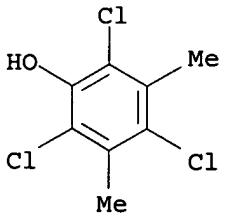
L11 ANSWER 1 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1962:416724 CAPLUS
DOCUMENT NUMBER: 57:16724
ORIGINAL REFERENCE NO.: 57:3345h-i,3346a-b
TITLE: Molecular compounds analogous to phenoquinone type
AUTHOR(S): Kumamoto, Sanetada
CORPORATE SOURCE: Univ. Kagoshima
SOURCE: Kogyo Kagaku Zasshi (**1961**), 64, 1812-16

CODEN: KGKZA7; ISSN: 0368-5462
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB Eight chlorinated and methylated p-quinones and twelve phenols, mainly chlorinated, were prepared These p-quinones (1 mole) and phenols (2 moles) were dissolved in benzene, CCl4, CHCl3, CS2, and left for several days. Several kinds of mol. compds. (I) were formed as crystals with beautiful colors, by evaporating the solvent. Among p-quinones, tetramethyl-p-benzoquinone was easiest to form I. However, the formation of I became more difficult as H atoms of the nucleus of p-benzoquinone were substituted with Cl atoms. Some of I were observed to change suddenly from their own proper color to yellow at their resp. discoloration temperature even below the m.p. This discoloration temperature is postulated as the temperature where the intermol. H bond is cut off with decomposition to individual constituents. The existence of intermol. H bond in the crystalline state was recognized by the infrared spectra. These crystals were mol. compds. analogous to phenoquinones formed by intermol. H bonds between p-quinones (1 mole) and phenols (2 moles).
IT **856302-22-2**, p-Benzoquinone, tetramethyl-, compound with 2,4,6-triohloro-3,5-xylenol **856303-09-8**, p-Benzoquinone, 2,5-dichloro-3,6-dimethoxy-, compound with 2,4,6-trichloro-3,5-xylenol **859803-13-7**, 3,5-Xylenol, 2,4,6-trichloro-, compound with trichlormethyl-p-benzoquinone **859803-19-3**, 3,5-Xylenol, 2,4,6-trichloro-, compound with methyl-p-benzoquinone **859803-22-8**, 3,5-Xylenol, 2,4,6-trichloro-, compound with 2,5-dimethyl-p-benzoquinone **859803-26-2**, 3,5-Xylenol, 2,4,6-trichloro-, compound with 2,5-dichloro-3,6-dimethyl-p-benzoquinone
(preparation of)

RN 856302-22-2 CAPLUS
CN p-Benzoquinone, tetramethyl-, compd. with 2,4,6-triohloro-3,5-xylenol (7CI) (CA INDEX NAME)

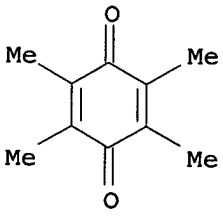
CM 1

CRN 6972-47-0
CMF C8 H7 Cl3 O



CM 2

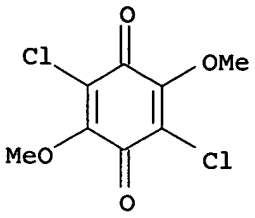
CRN 527-17-3
CMF C10 H12 O2



RN 856303-09-8 CAPLUS
CN p-Benzoquinone, 2,5-dichloro-3,6-dimethoxy-, compd. with 2,4,6-trichloro-3,5-xylenol (7CI) (CA INDEX NAME)

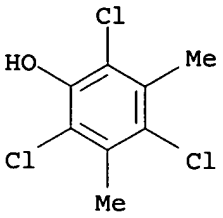
CM 1

CRN 7210-71-1
CMF C8 H6 Cl2 O4



CM 2

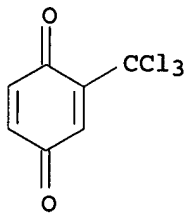
CRN 6972-47-0
CMF C8 H7 Cl3 O



RN 859803-13-7 CAPLUS
CN 3,5-Xylenol, 2,4,6-trichloro-, compd. with trichlormethyl-p-benzoquinone (7CI) (CA INDEX NAME)

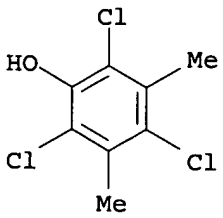
CM 1

CRN 856302-00-6
CMF C7 H3 Cl3 O2



CM 2

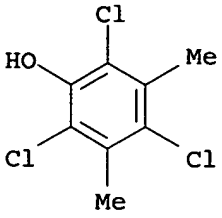
CRN 6972-47-0
CMF C8 H7 Cl3 O



RN 859803-19-3 CAPLUS
CN 3,5-Xylenol, 2,4,6-trichloro-, compd. with methyl-p-benzoquinone (7CI) (CA INDEX NAME)

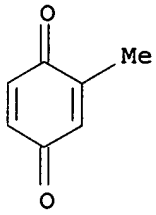
CM 1

CRN 6972-47-0
CMF C8 H7 Cl3 O



CM 2

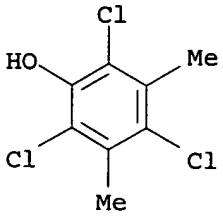
CRN 553-97-9
CMF C7 H6 O2



RN 859803-22-8 CAPLUS
CN 3,5-Xylenol, 2,4,6-trichloro-, compd. with 2,5-dimethyl-p-benzoquinone (7CI) (CA INDEX NAME)

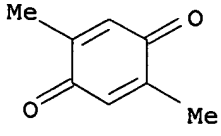
CM 1

CRN 6972-47-0
CMF C8 H7 Cl3 O



CM 2

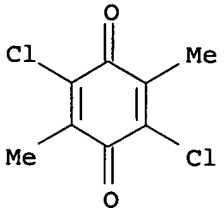
CRN 137-18-8
CMF C8 H8 O2



RN 859803-26-2 CAPLUS
CN 3,5-Xylenol, 2,4,6-trichloro-, compd. with 2,5-dichloro-3,6-dimethyl-p-benzoquinone (7CI) (CA INDEX NAME)

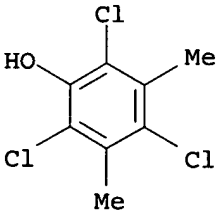
CM 1

CRN 46010-98-4
CMF C8 H6 Cl2 O2



CM 2

CRN 6972-47-0
CMF C8 H7 Cl3 O



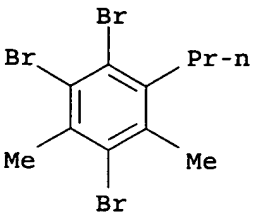
L11 ANSWER 2 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1956:69181 CAPLUS
DOCUMENT NUMBER: 50:69181
ORIGINAL REFERENCE NO.: 50:12856c-g
TITLE: The action of aluminum chloride on alkylbenzenes
AUTHOR(S): Nightingale, Dorothy V.; Shackelford, James M.
CORPORATE SOURCE: Univ. of Missouri, Columbia
SOURCE: Journal of the American Chemical Society (1956), 78, 1225-7
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB cf. C.A. 49, 14656e. 2,4-Me2C6H3COEt subjected to a Wolff-Kischner reduction yielded 68% 2,4-Me2C6H3Pr (I), b16 90-1°, nD25 1.4980; 2,4-Me2C6(NO2)3Pr (Ia), m. 108.5-109°; 2,4-Me2C6Br3Pr, oil. 2,4-Me2C6H3C(OH)Me2 reduced with hydrogen and Cu-Cr2O3 catalyst gave 2,4-Me2C6H3CHMe2 (II), b17 87-8°, nD20 1.4998; 2,4-Me2C6(NO2)3CHMe2 (III), m. 99-9.5°; 2,4-Me2C6Br3CHMe2, m. 63-4°. m-Xylene (IV) and iso-PrCl gave in the usual manner 1,3-Me2C6H3CHMe2 (V), b18 86-7°, nD25 1.4955, containing 13% 1,3,4-trialkylbenzene; 1,3-Me2C6(NO2)3CHMe2 (VI), m. 115.5-16°. IV (65 g.) and 31 g. AlCl3 treated slowly with stirring with 39 g. PrCl below 20°, and the mixture kept at room temperature overnight and decomposed with ice and HCl yielded 11 g. V, b. 80-6° (VI, m. 115-16°), 4 g. distillate, b. 86-8°, and 18 g. I, b. 88-91° (Ia, m. 98.5-99°). IV (200 cc.), 48 g. iso-PrOH, and 400 cc. 85% H2SO4 yielded 37 g. hydrocarbon, b15 85-6°, nD24 1.5004; the trinitro derivative, m. 177-8°, did not depress the m.p. of 1,3-Me2C6H(NO2)3. I (135 g.) treated with stirring on the steam bath with 29 g. AlCl3, the complex decomposed with iced HCl, and the hydrocarbon distilled yielded 22 g. IV, b16 75°, 5 g. mixed distillate, b16 85-9°, 35 g., b16

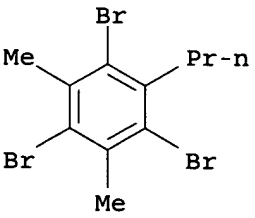
89-90°, nD22 1.4965 (III, m. 99-9.5°), 12 g. distillate, b16 90-1°, 12 g. distillate, b16 91-110°, and 20 g. distillate, b16 110-20°. II (55 g.) and 12 g. AlCl3 gave similarly 19 g. V containing 13% II. 1,2,4-C6H3Pr3 (VI) brominated yielded an oil, while p-C6H4Pr2 gave p-C6Br4Pr2, m. 107-8°. VI (29 g.) treated with 10 g. AlCl3 and the hydrocarbon fractionated gave a fraction, b. 130-40°, which contained mainly the 1,3,5-isomer of VI; 1,3,5-Pr3C6Br3, m. 112-13°; 1,3,5-Pr3C6(NO2)3, m. 123-4°.

IT **859785-24-3**, m-Xylene, 2,4,5-tribromo-6-propyl-
859785-26-5, m-Xylene, 2,4,6-tribromo-5-propyl-
(preparation of)

RN 859785-24-3 CAPLUS
CN m-Xylene, 2,4,5-tribromo-6-propyl- (5CI) (CA INDEX NAME)



RN 859785-26-5 CAPLUS
CN m-Xylene, 2,4,6-tribromo-5-propyl- (5CI) (CA INDEX NAME)



L11 ANSWER 3 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:	1956:27703 CAPLUS
DOCUMENT NUMBER:	50:27703
ORIGINAL REFERENCE NO.:	50:5542d-i,5543a-i,5544a-e
TITLE:	Additive compounds as possible intermediates in substitution processes. II
AUTHOR(S):	Bell, F.
CORPORATE SOURCE:	Heriot-Watt Coll., Edinburgh, UK
SOURCE:	Journal of the Chemical Society (1955) 2376-83 CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE:	Journal
LANGUAGE:	Unavailable

AB cf. C.A. 50, 898i. Chlorination of many sulfonanilides readily gave cyclohexene derivs. The reactions of these compds. were discussed. The halogenation of 3,4-xylidene (I) was studied. N-p-Toluenesulfonyl-p-toluidine (II) with Cl in either AcOH or CHCl3, or with SO2Cl2 alone or in CHCl3 gave 3,3,-5,6-tetrachloro-1-methyl-4-p-toluenesulfonyliminocyclohexene (III). Thus 10 g. II added slowly to excess SO2Cl2, the excess SO2Cl2 removed in vacuo, the residual oil dissolved in AcOH, and precipitated by addition of H2O gave 4.7 g. III, m. 184° (from AcOH, or C6H6). III reacted with piperidine but gave no definite compound Excess Zn dust added to a refluxing AcOH solution of III gave 2-chloro-N-(p-toluenesulfonyl)-toluidine (IIIa). III heated at 200° until gas evolution ceased gave 2,3,6-trichloro-N-(p-toluenesulfonyl)-p-toluidide (IV), m. 160° (from AcOH). IV in cold H2SO4 gave 2,3,6-trichloro-p-toluidine (V) almost quantitatively, m. 60-2°; Ac derivative, m. 196°; di-Ac derivative, m. 150°. III reacted vigorously with C5H5N and on acidification gave a poor yield of IV. III (2 g.) heated 1.5 hrs. at 150° with 4

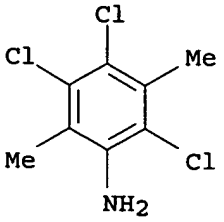
cc. H_2SO_4 and 4 cc. H_2O gave a black tar, and addition of excess of aqueous NH_3 yielded V. Addition of $PhNH_2$ to III gave an impure solid, m. 135°. III dissolved during 3 hrs. in refluxing EtOH gave 1,1,3,4-tetrachloro-6(?)-ethoxy-5-methyl-2-p-toluenesulfonyliminocyclohexane, m. 195°, and IV. A similar reaction with MeOH gave the MeO analog (VI), m. 187° (from EtOH). Reduction of VI by Zn dust and AcOH gave 2-chloro-N-(p-toluenesulfonyl)-p-toluidine, m. 220° (decomposition), and some IV. Solution in warm C_5H_5N gave almost quant. conversion to 2,6-dichloro-3-methoxy-N-(p-toluenesulfonyl)-p-toluidine (VII) m. 190° (from AcOH). VII was also obtained by use of $PhNH_2$ but was accompanied by sticky, red decomposition products. Aceto-p-toluidide (VIII) chlorinated by Cohen and Dakin's method [J. Chemical Society 81, 1337(1902)] gave a crude product, m. about 179°, which on hydrolysis in EtOH-HCl gave a base, m. 35-47°. This was purified and reacetylated to yield the Ac and di-Ac derivs. of V. With p-tosyl chloride it gave IV. Similarly 2,3,6-trichloro-N-benzenesulfonyl-p-toluidine (IX) was obtained, m. 169° (from EtOH). In a typical experiment 5 g. VIII gave 1.9 g. of the 2,6-dinitro derivative, m. 190°. Various proportions of HNO_3 and HCl were used but in no case was any of the trichloro derivative isolated. 2,6-Dichloro-p-toluidine gave the N-benzenesulfonyl derivative, m. 169° (from AcOH), and the N-(p-toluenesulfonyl) derivative, m. 180° (from C_6H_6). IIIa in C_5H_5N on treatment with N-bromosuccinimide gave 2-bromo-6-chloro-N-(p-toluenesulfonyl)-p-toluidine, m. 180°. It was unchanged after treatment with Zn dust in refluxing AcOH. II in $CHCl_3$ treated with 1 mole Br gave the 2-bromo derivative (X), m. 118° (from EtOH). II in C_5H_5N with 2 moles N-bromosuccinimide yielded 2,6-dibromo-N-(p-toluenesulfonyl)-p-toluidine (XI), m. 179-80° (from AcOH). X and XI did not react with Zn dust in AcOH. p-Toluidine treated with Cl in AcOH gave thick, dark oils. N-Benzenesulfonyl-p-toluidine (XII) in $CHCl_3$ treated with excess Cl yielded 4-benzenesulfonylimino-3,3,5,6-tetrachloro-1-methylcyclohexene (XIII), m. 164°. XIII reacted vigorously with C_5H_5N to give IX in small yield. XIII with $PhNH_2$ gave a mixture from which was obtained a powder, m. 158° (decomposition) (from AcOH), which appeared to have been produced by loss of HCl from XIII. The o-isomer (XIV) of XII (4 g.) was similarly chlorinated to give 6-benzenesulfonylimino-3,3,4,5-tetrachloro-1-methylcyclohexene (XV), m. 166° (from AcOH). The yield of XV was not improved by using N-benzenesulfonyl-4-chloro-o-toluidine (XVI) as starting material. XV treated with Zn in AcOH yielded XVI. XV with $PhNH_2$ gave 4-anilino-N-benzenesulfonyl-5,6-dichloro-o-toluidine, m. 207° (from AcOH), and was hydrolyzed by cold H_2SO_4 to 4-anilino-5,6-dichloro-o-toluidine, m. 115° (from EtOH). XV heated above the m.p. gave N-benzenesulfonyl-4,5,6-trichloro-o-toluidine,m. 159°. N-p-Toluene sulfonyl-o-toluidine (XVII) (14 g.) submitted to the same process gave 4 g. 4-chloro derivative (XVIII) and an oil. N-Acetyl-2-chloro-p-toluidine gave 1 g. 2,6-dichloro amide and an uncrystallizable oil. When excess SO_2Cl_2 wsa added to the sulfonamide, and after the initial reaction, excess SO_2Cl_2 removed and the residue dissolved in EtOH or AcOH, the following results were obtained: XVII (5 g.) gave 4 g. XVIII, m. 144°; XIV (4 g.) gave 2.6 g. 4-chloro derivative and a small yield of XV; N-benzenesulfonyl-m-toluidine gave an almost quant. yield of the dichloro derivative, m. 116°; N-p-toluenesulfonyl-m-toluidine gave crude 2,4-dichloro-N-p-toluenesulfonyl-m-toluidine (XIX), pure XIX, prepared from 2,4-dichloro-p-toluidine, m. 145° (from AcOH); XII (5 g.) gave 2.5 g. 2-Cl derivative and an oil; N-p-toluenesulfonyl-p-anisidine gave the dichloro derivative (XX), m. 166°, XX was hydrolyzed to the dichloro base, m. 75°. 2,3-Xylidine with p-tosyl chloride in C_5H_5N yielded N-p-toluenesulfonyl-2,3-xylidine (XXI), m. 147° (from EtOH). XXI in $CHCl_3$ treated with Cl gas gave an almost quant. yield of 4-chloro-N-p-toluenesulfonyl-2,3-xylidine (XXII), m. 142° (from EtOH). XXII, alternatively prepared from 4-chloro-2,3-xylidine and p-tosyl chloride in C_5H_5N, was unchanged after 2 days in concentrated H_2SO_4. XXI with excess SO_2Cl_2 gave 4,6-dichloro-N-p-toluenesulfonyl-2,3-xylidine, m. 174°, and a main crop of 3,3,4,5-tetrachloro-1,2-dimethyl-6-p-toluenesulfonyliminocyclohexene (XXIII), m. 128-30°. XXIII refluxed 3 hrs. in EtOH, by solution in C_5H_5N, or at 190° gave 4,5,6-trichloro-N-p-toluenesulfonyl-2,3-xylidine, m. 206° (from AcOH). Reduction of XXIII in AcOH with Zn dust gave XXII, and with $PhNH_2$

it reacted to give 4-anilino-5,6-dichloro-N-p-toluenesulfonyl-2,3-xylidine, m. 205° (from AcOH) Cl passed into N-p-toluenesulfonyl-3,4-xylidine (XXIV) in CHCl3 gave a small crop of unchanged XXIV and an oil. When XXIV (5 g.) was treated with SO2Cl2 it yielded 0.6 g. putative 3,3,5,5,6-pentachloro-4-p-toluenesulfonylimino-1,2-dimethylcyclohexene (XXV), m. 187° (decomposition) (from AcOH). XXV yielded an uncrystallizable material on thermal decomposition, and on reduction with Zn in AcOH a complex mixture N-Acetyl-3,4-xylidine (XXVI) on chlorination yielded a monochloro derivative, m. 153°. Passage of excess Cl into a CHCl3 solution of XXVI led to oils. 2,4-Xylidine with p-tosyl chloride gave N-p-toluenesulfonyl-2,4-xylidine (XXVII), m. 109° (from EtOH). XXVII with excess SO2Cl2 gave a low yield of a trichloro derivative (XXVIII), m. 163° (decomposition) (from AcOH). Cl passed into a CHCl3 solution of XXVII gave a small amount of XXVIII and a small crop of 3,5,6-trichloro-N-p-toluenesulfonyl-2,4-xylidine, m. 152° (from AcOH). Commercial NaOCl solution added to XXVII in AcOH at 50° yielded 6-chloro-N-p-toluenesulfonyl-2,4-xylidine, m. 158°. N-Benzenesulfonyl-2,4-xylidine (XXIX) (8 g.) with SO2Cl2 yielded a somewhat impure trichloro derivative with highest m.p. at 198-202°. Chlorination of XXIX in CHCl3 gave similar results. p-Toluenesulfonyl-2,5-xylidine (XXX) similarly treated with SO2Cl2 and the oil remaining after removal of the solvent taken up in AcOH gave crop A and the filtrate treated with H2O gave a gummy product. This gum upon repeated crystallization from EtOH gave a tetrachloro derivative (XXXI), m. 152°. Reduction of XXXI by Zn dust and AcOH gave 4-chloro-N-p-toluenesulfonyl-2,5-xylidine (XXXII), m. 145° (from EtOH). XXXI vigorously reacted with C5H5N but gave no recognizable product as did treatment with cold concentrated H2SO4. Crop A yielded putative 3,3,5-trichloro-6-p-toluenesulfonamido-1,4-dimethylcyclohexa-1,4-diene (XXXIII), m. 132°. Solution of XXXIII in hot EtOH gave 3,4,6-trichloro-N-p-toluenesulfonyl-2,5-xylidine (XXXIV), m. 167°, also obtained in small yield from the products of thermal decomposition of, and on suspension of XXXIII in cold concentrated H2SO4. Upon addition of XXXIII to C5H2N, the solution became momentarily bright yellow and then faded; addition of dilute HCl gave 4,6-dichloro-N-p-toluenesulfonyl-2,5-xylidine (XXXV), m. 125-6° (from EtOH). XXXV gave the dichloro base on hydrolysis, m. 166-8°, and this without further purification was acetylated to N-acetyl-4,6-dichloro-2,5-xylidine (XXXVI), m. 166-8° (from EtOH), alternatively prepared from N-acetyl-2,5-xylidine and SO2Cl2. Reduction of XXXIII in refluxing AcOH with Zn dust produced a material, m. 117-20°, apparently a mixture of mono- and trichloro derivs., for after dissolution in H2SO4 there was obtained XXXII. XXXIV was not reduced with Zn dust in refluxing AcOH, but on dissolution in H2SO4 readily gave the amine, m. 206°. Addition of SO2Cl2 to XXX gave an almost theoretical yield of XXXII. Cl passed into a solution of 5 g. XXX in CHCl3 gave 1.2 g. crude XXXII and 1 g. XXXIII. XXX (2 g.) treated with excess NaOCl in AcOH at 50° yielded XXXII contaminated with unchanged material. Cl passed into a solution of N-p-toluenesulfonyl-2,6-xylidine (XXXVII) in CHCl3 gave the monochloro derivative (XXXVIII), m. 158° (from AcOH or EtOH). XXXVII (4 g.) with excess SO2Cl2 gave 1.9 g. XXXVIII. XXVI in AcOH treated with Br gave after 24 hrs. a product which was essentially N-acetyl-6-bromo-3,4-xylidine (XXXIX), m. 164° (from EtOH). Hydrolysis of XXXIX with refluxing EtOH-HCl gave 6-bromo-3,4-xylidine; p-toluenesulfonyl derivative (XL), m. 122° (from EtOH). Addition of Br in CHCl3 to a CHCl3 solution of XXIV gave XL in almost quant. yield. Heating XXIV in CHCl3 with Br gave XL together with the HBr salt of 2,6-dibromo-3,4-xylidine. Addition of Br to XXIV in cold C5H5N gave the 2,6-dibromo derivative (XLI), m. 165° (from EtOH). Dissolution of XLI in cold H2SO4 gave the 2,6-dibromo amine; Ac derivative (XLII), m. 198°. XLII crystallized from EtOH as needles, which in contact with solvent were slowly transformed into prisms. Passage of Cl into either XXXIX or XL in CHCl3 led to no crystalline product other than the initial compds. XL was unchanged after treatment with iodine in C5H5N.

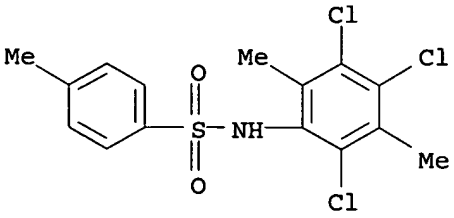
IT **857974-28-8**, 2,5-Xylidine, 3,4,6-trichloro- **859493-05-3**, p-Toluenesulfono-2',5'-xylidide, 3',4',6'-trichloro- **859493-26-8**, p-Toluenesulfono-2',3'-xylidide, 4',5',6'-trichloro- **859791-74-5**, p-Toluenesulfono-2',4'-xylidide, 3',5',6'-trichloro-
(preparation of)

RN 857974-28-8 CAPLUS

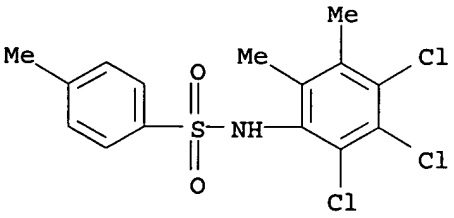
CN 2,5-Xylidine, 3,4,6-trichloro- (5CI) (CA INDEX NAME)



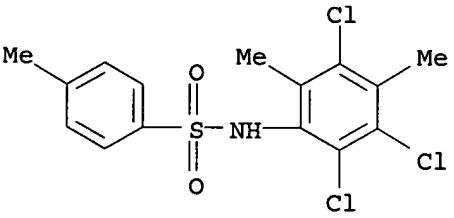
RN 859493-05-3 CAPLUS
CN p-Toluenesulfono-2',5'-xylidide, 3',4',6'-trichloro- (5CI) (CA INDEX NAME)



RN 859493-26-8 CAPLUS
CN p-Toluenesulfono-2',3'-xylidide, 4',5',6'-trichloro- (5CI) (CA INDEX NAME)



RN 859791-74-5 CAPLUS
CN p-Toluenesulfono-2',4'-xylidide, 3',5',6'-trichloro- (5CI) (CA INDEX NAME)



L11 ANSWER 4 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1955:77678 CAPLUS
DOCUMENT NUMBER: 49:77678
ORIGINAL REFERENCE NO.: 49:14656e-i,14657a-b
TITLE: The action of aluminum chloride on alkylbenzenes. IV
AUTHOR(S): Nightingale, Dorothy V.; Shackelford, James M.
CORPORATE SOURCE: Univ. of Missouri, Columbia
SOURCE: Journal of the American Chemical Society (**1954**), 76, 5767-70
CODEN: JACSAT; ISSN: 0002-7863

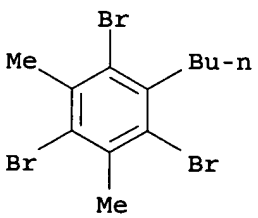
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB cf. C.A. 49, 228a. When 2,4-Me2C6H3Bu (I), 2,4-Me2C6H3CHEtMe (II), and 2,4-Me2C6H3CH2CHMe2 (III) are warmed with AlCl3, the C4H9 group migrates to the 5-position without isomerization. The alkylation of m-xylene (IV) with BuCl yielded a mixture of 3,5-Me2C6H3Bu (V) and 3,5-Me2C6H3CHEtMe (VI). The alkylation of IV with EtMeCHCl yielded VI. BuCl (72 g.) added slowly with stirring to 127 g. IV and 60 g. AlCl3 below 35°, the mixture kept at room temperature overnight, decomposed with iced HCl, extracted with Et2O, and the extract washed, dried, and distilled yielded 20 g. VI, b15 96-7°, n25D 1.4958 (trinitro derivative, m. 90-1°), and 35 g. V, b15, 103-4°,n25D 1.4947 (trinitro derivative, m. 93-4°). IV (180 g.) alkylated in the same way with 110 g. EtMeCHCl and 95 g. AlCl3 yielded 125 g. VI, b15 95-7°, n25D 1.4929 (trinitro derivative, m. 90-1°). IV (250 cc.) and 61 cc. EtMeCHOH treated slowly with stirring with 330 cc. concentrated H2SO4 and 70 cc. H2O, the mixture stirred 7 h. at room temperature, and the hydrocarbon layer washed, dried, and distilled gave 68 g. trialkylbenzene, b28 106-8°, n24D 1.4967, which on nitration yielded only the trinitro derivative of IV, m. 181-2°. The trialkylbenzene (35 g.) warmed 3.5 h. with 8.5 g. AlCl3 and distilled gave 5 g. distillate b17 88-94°, and 12 g. b17 94-7°; the nitration product from the 2nd fraction, recrystd. repeatedly, yielded a small amount of the trinitro derivative of VI. PrMgBr from 98 g. PrBr in 200 cc. Et2O distilled to remove 100 cc. Et2O, the residual solution diluted with 100 cc. C6H6, treated as rapidly as possible with 160 g. CdCl2, refluxed 1 h., the mixture treated with 72 g. 3,5-Me2C6H3COCl (VII), refluxed 1 h., decomposed in the usual manner, extracted with Et2O, and the C6H6-Et2O solution washed with aqueous Na2CO3 and H2O, dried, and distilled yielded 31 g. 3,5-Me2C6H3COPr (VIII), b16 139°, n25D 1.5169; semicarbazone, m. 157°. VIII reduced with H and CuO-Cr2O3 catalyst gave V, b15 102-4°, n25D 1.4937 (trinitro derivative, m. 95°). VII and Et2Cd yielded similarly 48% 3,5-Me2C6H3COEt (IX), b15 110°, n25D 1.5143; semicarbazone, m. 167°. IX (45 g.) and MeMgI yielded in the usual manner 24 g. 3,5-Me2C6H3C(OH)EtMe, b15 109-11°, n24D 1.5148, which, hydrogenated over CuO-Cr2O3, yielded 21 g. VI, b16, 95-6°, n24D 1.4929; trinitro derivative, granules from EtOH, m. 91°. AlCl3 (23 g.) added to 102 g. I, the mixture warmed on the steam bath 3.5 h., decomposed with iced HCl, and the product isolated in the usual manner yielded 31 g. V, b16 103-5° (trinitro derivative, m. 94°; tri-Br derivative, m. 53°). II (133 g.) and 29 g. AlCl3 gave similarly VI in 2 fractions, b16 91-5° (18 g.), and b16 95-6° (63 g.); trinitro derivative, granules, m. 91°. III (100 g.) and 20 g. AlCl3 gave in the same way 20 g. product, b17 98-101°, which yielded a trinitro derivative m. 85-6° (either the derivative of 3,5-Me2C6H3CH2CHMe2 or a eutectic mixture). The trinitro derivs. of the hydrocarbons were prepared by adding 2 cc. hydrocarbon slowly with shaking to 10 cc. ice-cold concentrated H2SO4 and 5 cc. fuming HNO3, warming the mixture on the water bath at 60-80°, pouring onto ice, and washing and recrystg. the precipitate from EtOH; in this manner were prepared the trinitro derivs. of III, m. 115-16°, and of 3,5-Me2C6H3CMe3, m. 86° with softening at 80-5°. The brominations were carried out with liquid Br in the presence of Fe powder.

IT **859781-62-7**, m-Xylene, 2,4,6-tribromo-5-butyl-
(preparation of)

RN 859781-62-7 CAPLUS

CN m-Xylene, 2,4,6-tribromo-5-butyl- (5CI) (CA INDEX NAME)



L11 ANSWER 5 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN

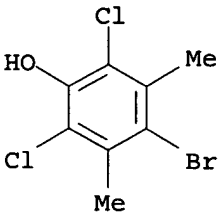
ACCESSION NUMBER: 1955:35841 CAPLUS
DOCUMENT NUMBER: 49:35841
ORIGINAL REFERENCE NO.: 49:6871f-h
TITLE: 2,6-Dichloro-3,5-xylenol
AUTHOR(S): Gump, Wm. S.; Nikawitz, Edward J.
CORPORATE SOURCE: Givaudan Corp., Delawanna, NJ
SOURCE: Journal of the American Chemical Society (**1953**), 75, 6311-12
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 49:35841

AB Direct chlorination of 3,5-xylenol or 2-chloro-3,5-xylenol yields mixts. containing mostly the 2,4-dichloro derivative (I); benzoate, m. 114-15°. 4-Bromo-3,5-xylenol (78 g.) in 1.5 l. CCl4 treated during 10 hrs. with 150 g. Cl at 70-5°, the solution poured into water, the organic layer evaporated, the residue (120 g.) extracted by refluxing 1 hr. with 600 cc. water containing 100 g. NaOH, followed by 3-30 min. extns. with 20 g. NaOH in 500 cc. water, and the combined ests. filtered and acidified with HCl yielded 46.1 g. 4-bromo-2,6-dichloro-3,5-xylenol (II), m. 183-4°. II(34 g.) and 600 cc. 20% KOH heated to 90°, the solution treated during 2 hrs. with 80 g. Zn dust, heated 3 hrs. at 90°, filtered, the cold filtrate acidified with HCl (ice cooling), filtered, the product refluxed with 100 cc. EtOH containing C, the filtrate diluted with water, and the precipitate (26 g.) distilled yielded 18.3 g. 2,6-dichloro-3,5-xylenol (III), b1 105-10°, m. 87-8°; benzoate, m. 143-5°. I and III have an unpleasant odor. III is much less active bactericidally than I.

IT **408314-44-3**, 3,5-Xylenol, 4-bromo-2,6-dichloro-
(preparation of)

RN 408314-44-3 CAPLUS

CN Phenol, 4-bromo-2,6-dichloro-3,5-dimethyl- (9CI) (CA INDEX NAME)



L11 ANSWER 6 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1954:10924 CAPLUS
DOCUMENT NUMBER: 48:10924
ORIGINAL REFERENCE NO.: 48:1989d-g
TITLE: Chloramines as a source of iodine chloride. Preparation of iodophenols, -naphthols and -aromatic ethers by means of a chloramine and an iodide
AUTHOR(S): Jones, Brynmor; Richardson, Eileen N.
CORPORATE SOURCE: Univ. Coll., Hull, UK
SOURCE: Journal of the Chemical Society (**1953**) 713-15
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

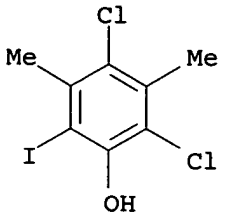
AB The reagent (dichloramine-T and NaI) was added cautiously to a well-stirred solution of the phenol or ether in HOAc at room temperature, except for hydroxy and alkoxynaphthoic acids which reacted at 60-75°. With exception of o- and p-C6H4(OMe)2, the yields were 75-92%. Below are listed the products (and their m.ps.) obtained by this method: p-IC6H4OMe, 52°; p-IC6H4OEt, 27°; 1,2-IC10H6OMe, 88°; 1,2-IC10H6OEt, 74°; 4,1-IC10H6OEt, 45°; 6,2,4-ICl2C6H2OH, 62°; 6,2,4-IBr2C6H2OH, 104°; 5-iodo-3-nitro-p-cresol, 83°; 2-chloro-4-iodo-m-5-xylenol, 90°; 2-chloro-4,6-diiodo-m-5-xylenol, 131°; 2,4-dichloro-6-iodo-m-5-xylenol, 130°; 3,5-diiodosalicylic acid, 233°; 4-hydroxy-3,5-diiodobenzoic acid,

262°; 3-hydroxy-4-iodo-2-naphthoic acid, 210° (decomposition); 6-hydroxy-5-iodo-2-naphthoic acid, 234°; 5-iodo-6-methoxy-2-naphthoic acid, 292° (decomposition); 5-iodo-6-lauroyloxy-2-naphthoic acid, mesomorphic, smectic phase 145-67°, nematic phase 167-70°; 8-iodo-7-octyloxy-2-naphthoic acid, 148°; 7-cetyloxy-8-iodo-2-naphthoic acid, 123°; 4,6-diiodo-1,3-dimethoxybenzene, 75°; 4,5-diiodo-1,2-dimethoxybenzene, 134° (30% yield); 2,5-diiodo-1,4-dimethoxybenzene, 171° (20% yield); 2,4-diiodo-3,5-dimethoxybenzene, 125° (50% yield).

IT **791626-88-5**, 3,5-Xylenol, 2,4-dichloro-6-iodo- **791626-94-3**, 3,5-Xylenol, 4-chloro-2,6-diiodo-
(preparation of)

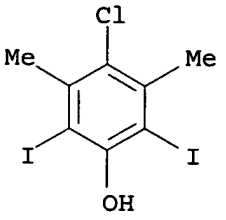
RN 791626-88-5 CAPLUS

CN 3,5-Xylenol, 2,4-dichloro-6-iodo- (5CI) (CA INDEX NAME)



RN 791626-94-3 CAPLUS

CN 3,5-Xylenol, 4-chloro-2,6-diiodo- (5CI) (CA INDEX NAME)



L11 ANSWER 7 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1950:34747 CAPLUS

DOCUMENT NUMBER: 44:34747

ORIGINAL REFERENCE NO.: 44:6645h-i,6646a-e

TITLE: Halogenated anthraquinone dyes

PATENT ASSIGNEE(S): Sandoz Ltd.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CH 259730		19490701	CH	

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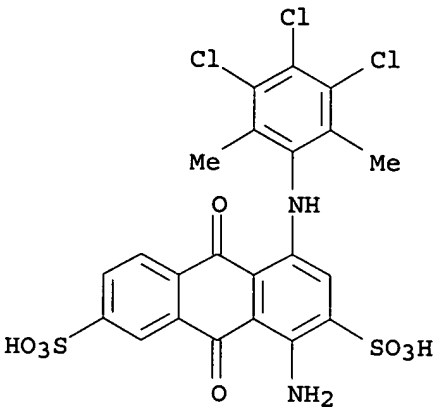
AB Halogenation of 1-amino-4-anilinoanthraquinonedisulfonic acids is described. The products dye wool, silk, and nylon in bright reddish blue shades. K 1-amino-4-(2,4,6-trimethylanilino)-2,6-anthraquinonedisulfonate (I) 5.9 g. is dissolved at 25° in 85% H_2SO_4 50 and chlorinated at 20-30° with Cl 2. The mixture is heated 1 h. at 60° and drowned in ice and H_2O to obtain the reddish blue crystals of H_2O-soluble dye, 1-amino-4-(3,5-dichloro-2,4,6-trimethlanilo)-2,6-anthraquinonedisulfonic acid. In Swiss 259,731, K 1-amino-4-(2,6-dimethylanilino)-2,8-anthraquinonedisulfonate 5.8 is brominated in 90% H_2SO_4 50 with Br 1.8 at 40° for 3 h. to give 1-amino-4-(dibromo-2,6-dimethylanilino)-2,8-anthroquinonedisulfonic acid. In Swiss 259,732, Na 1-amino-4-(2,6-diethylanilino)-2,7-anthraquinonedisulfonate, brominated as above at 60° for 3 h. gave 1-amino-4-(dibromo-2,6-di-ethylanilino)-2,7-anthraquinonedisulfonic acid. In Swiss 259,733, Na

1-amino-4-(2',4',6-trimethylanilino)-2,5-anthraquinonedisulfonate 5.6 is brominated in 100% H_2SO_4 30 with Br 1.8 by stirring at 20-30° overnight and heating if necessary to 100° for completion. The crystals of 1-amino-4-(3,5-dibromo-2,4,6-trimethylanilino)-2,5-anthraquinonedisulfonic acid (II) are reddish blue. In Swiss 259,734, I 5.9 is brominated in 90% H_2SO_4 30 by stirring overnight at 20-30° and heating as required to 100° for completion to give the 2,6-isomer of II. In Swiss 259,735, Na 1-amino-4-(2,4,6-triethylanilino)-2,8-anthraquinonedisulfonate 6 is brominated in 100% H_2SO_4 30 with Br 1.8 at 20-30° and then heated if necessary to 60° for completion to give 1-amino-4-(3,5-dibromo-2,4,6-triethylanilino)-2,8-anthraquinonedisulfonic acid (III). In Swiss 259,736, 1-amino-2-bromo-4-(2,6-dimethyl-4-chloroanilino)-7-anthraquinonesulfonic acid 5 in 1-2% oleum 25 is treated with iodine 0.05 and Cl 2. The dye is isolated as before and converted to the 2,7-disulfonic acid by replacement of the -Br with -SO_3H using K_2SO_3 under pressure. The dye is 1-amino-4-(3,4,5-trichloro-2,6-dimethylanilino)-2,7-anthraquinonedisulfonic acid. In Swiss 259,737, Na 1-amino-2-bromo-4-(2,4,6-triethylanilino)-6-anthraquinonesulfonate 5.8 is brominated in 90% H_2SO_4 30 with Br 2. After stirring overnight at 20-30° the temperature is raised to 100° for 1-2 h. for completion. This dye is converted as above to the disulfonate using K_2SO_3 under pressure to give the 2,6-isomer of III. In Swiss 259,738, Na 1-amino-4-(2,3,6-triethylanilino)-2,5-anthraquinonedisulfonate is brominated at 40° for 3 h. in 95% H_2SO_4 50 with Br 1. 1-Amino-4-(bromo-2,3,6-triethylanilino)-2,5-anthraquinonedisulfonic acid is obtained in the form of reddish-blue needles.

IT **737803-95-1**, 2,7-Anthraquinonedisulfonic acid, 1-amino-4-(3,4,5-trichloro-2,6-xylidino)-
(preparation of)

RN 737803-95-1 CAPLUS

CN 2,7-Anthraquinonedisulfonic acid, 1-amino-4-(3,4,5-trichloro-2,6-xylidino)- (5CI) (CA INDEX NAME)



L11 ANSWER 8 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1948:38890 CAPLUS

DOCUMENT NUMBER: 42:38890

ORIGINAL REFERENCE NO.: 42:8255e-i

TITLE: The effect of Tween 80 in vitro on the bacteriostatic activity of twenty compounds for Mycobacterium tuberculosis

AUTHOR(S): Youmans, Anne S.; Youmans, Guy P.

CORPORATE SOURCE: Northwestern Univ. Med. School, Chicago

SOURCE: Journal of Bacteriology (**1948**), 56, 245-52
CODEN: JOBAAY; ISSN: 0021-9193

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

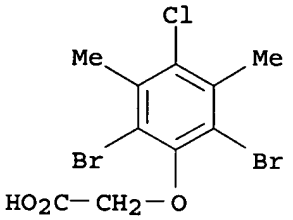
AB Unpurified Tween 80 in a synthetic medium, as compared to the medium alone, increased the activity of the following compds.: sulfanilamide; sulfapyridine; sulfathiazole; 4-allylaminophenyl 4-aminophenyl sulfone;

2-nitro-5-sulfanilylthiophene; di(2-nitro-5-thienyl) sulfone; di(4-aminophenyl) sulfone; p-xyloquinone; 1,4-naphthoquinone; 2-chloro-1,4-naphthoquinone; 8-hydroxyquinoline; (2,6-dibromo-3,5-dimethyl-4-chlorophenoxy)acetic acid; 4-amino-2-hydroxybenzoic acid-HCl; 3-indolepropionic acid; and chloromycetin. It decreased the activity of: p-aminoazobenzene; 2-(4-aminophenyl)pyridine; and 4-chlorophenyl 4-aminophenyl sulfide. It had no effect on: 2-methylnaphthoquinone and N-(p-aminophenyl)piperidine. Crystalline bovine albumin increased the activity of 13 out of the 20 compds. If Tween and albumin were both present; 5 compds. were more bacteriostatic, 4 gave approx. the same results as the basal medium, and 11 were less bacteriostatic. Purified Tween had less effect than unpurified Tween. Decreasing the concentration of Tween by 4-fold did not alter significantly the bacteriostatic levels of 5 compds. studied. The free oleic acid which may be present in Tween apparently was not responsible for the effect of Tween on the bacteriostatic activities of the compds. mentioned. 17 references.

IT **764710-09-0**, Acetic acid, (2,6-dibromo-4-chloro-3,5-xylyloxy)- (bacteriostatic activity for Mycobacterium tuberculosis)

RN 764710-09-0 CAPLUS

CN Acetic acid, (2,6-dibromo-4-chloro-3,5-dimethylphenoxy)- (9CI) (CA INDEX NAME)



L11 ANSWER 9 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1948:23164 CAPLUS

DOCUMENT NUMBER: 42:23164

ORIGINAL REFERENCE NO.: 42:4957h-i,4958a-e

TITLE: Halogenation of m-5- and m-2-xylenols. Preparation and structure of certain polyhalo-m-5- and -m-2-xylenols

AUTHOR(S): Elston, C. H. R.; Peters, A. T.; Rowe, F. M.

CORPORATE SOURCE: Univ. of Leeds, UK

SOURCE: Journal of the Chemical Society (**1948**) 367-70
CODEN: JCSOA9; ISSN: 0368-1769

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB 2,4,6-Br3C6H2OH (I) (16 g. as a paste with 20 cc. H2O), treated with 16 g. Cl in 2000 cc. H2O at room temperature, gives 2,4,6-tribromophenol chloride (II), yellow, m. 125-30° (decomposition); the alc. solution of II, decolorized with SO2, gives a mixture of I and 4,2,6-ClBr2C6H2OH (III). III (14.3 g.) and 8.8 g. Br in dilute AcOH give 4-chloro-2,6-dibromophenol bromide, pale yellow, m. 99-100°, decompose 120°; SO2 in EtOH gives only III. 2,4,6,3,5-Br3Me2C6OH (IV) in AcOH, treated with 1 mol. Cl below 30°, the solution poured into H2O, the wet product taken up in petr. ether at 30-40°, and the residue quickly crystallized from petr. ether, gives 2-chloro-2,4,6-tribromo-1,3-dimethyl-3,6-cyclohexadien-5-one, m. 120-2°; decomposition with SO2 yields mainly 2,4,6,3,5-ClBr2Me2C6OH (V); the action of 2.5 mols. Cl on V in AcOH at 10° and the mixture allowed to stand overnight gives 2,2,4-trichloro-6-bromo-1,3-dimethyl-3,6-cyclohexadien-5-one, very pale yellow, m. 105°, moderately stable when dry but the crude product decompose readily; SO2 in EtOH gives 2,6,4,3,5-Cl2BrMe2C6OH, m. 165-6°. Br does not react with V but with 2,4,6,3,5-Cl3Me2C6OH (VI) in AcOH at 60° (with heating 2 min. at 110° and standing overnight at room temperature) it yields 2,4,6-trichloro-3,5-xylenol bromide, yellow, m. 112°; it decompose rapidly in air and on boiling with EtOK it gives VI. 4,6,2,3,5-Cl2BrMe2C6OH in AcOH, treated at 40-50° with 1.2 mols. Cl, gives about 4 parts 2,2,4,6-tetrachloro-1,3-dimethyl-3,6-cyclohexadien-5-one

(VIA) (m. 107-8°) and 1 part of a compound m. 106-7° (mixed m.p. 70-90°), stable to SO2 in aqueous EtOH, whose constitution was not established. 2,6,3,4,5-Me2Br3C6OH (VII) and 2 mols. Br in H2O give 85.3% 3,4,5-tribromo-2,6-xylenol bromide (VIII), yellow, m. 139-40°; decomposition of VIII with boiling EtOH (more readily with SO2 in EtOH) gives VII; Zn and H2O gives VII and some 4,5-di-Br derivative; boiling aqueous KI gives scarlet needles, m. 156-8°, of the adduct of 1 mol. 4,6-dibromo-m-xyloquinone and 2 mols. VIII. VII and 2.1 mols. Cl in AcOH, warmed 2 min. at 60° and kept 2 hrs. at room temperature, give 3,4,5-tribromo-2,6-xylenol chloride, yellow, m. 60-1°; prolonged boiling with EtOH gives VII. The only products isolated from 4,3,5,2,6-ClBr2Me2C6OH (IX) and Br were 4,6-dibromo-m-xyloquinone (X) and an adduct of 1 mol. X and 2 mols. IX, m. 138-40°. Absorption spectra are given for PhOH, VI, VIA, C6H4O2, tetrabromo-2,6-xylenol, and II; the data support the quinonoid structure of the polyhalo compds.

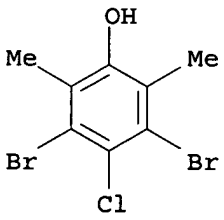
IT **857974-71-1**, 2,6-Xylenol, 3,5-dibromo-4-chloro-, compound with 3,5-dibromo-m-xyloquinone **859783-89-4**, 3,5-Xylenol, 2,4,6-trichloro-, hypobromite **874519-00-3**, 3,5-Xylenol, 2-bromo-4,6-dichloro-
(preparation of)

RN 857974-71-1 CAPLUS

CN m-Xyloquinone, 3,5-dibromo-, compd. with 3,5-dibromo-4-chloro-2,6-xylenol (5CI) (CA INDEX NAME)

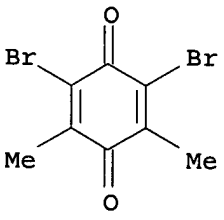
CM 1

CRN 408326-71-6
CMF C8 H7 Br2 Cl O



CM 2

CRN 87405-27-4
CMF C8 H6 Br2 O2



RN 859783-89-4 CAPLUS

CN 3,5-Xylenol, 2,4,6-trichloro-, hypobromite (5CI) (CA INDEX NAME)

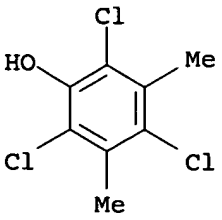
CM 1

CRN 13517-11-8
CMF Br H O

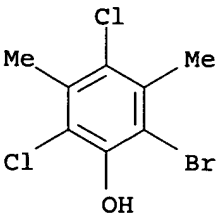
Br—OH

CM 2

CRN 6972-47-0
CMF C8 H7 Cl3 O



RN 874519-00-3 CAPLUS
CN 3,5-Xylenol, 2-bromo-4,6-dichloro- (5CI) (CA INDEX NAME)



L11 ANSWER 10 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1948:21313 CAPLUS
DOCUMENT NUMBER: 42:21313
ORIGINAL REFERENCE NO.: 42:4548h-i,4549a-d
TITLE: Halogenation of 3,5- and 2,6-xylenol. Mixed chlorobromo derivatives
AUTHOR(S): Gleed, S. W.; Peters, A. T.
CORPORATE SOURCE: Univ. Leeds, UK
SOURCE: Journal of the Chemical Society (**1948**) 209-11
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 42:21313

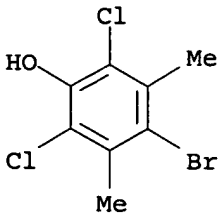
AB 1,3,2,5-Me2ClC6H2OH (I) (20 g.) in 200 cc. CHCl3 containing a little anhydrous AlBr2, treated rapidly with 20.5 g. Br in 100 cc. CHCl3, gives 96.3% 4-chloro-2-bromo-3,5-xylenol (II), m. 68°. 1,3,2,5-Me2BrC6H2OH (III) (20 g.) in CHCl3 containing a little iodine, treated rapidly with 7.1 g. Cl in 100 cc. CHCl3, gives 77-4% 2-chloro-4-bromo-3,5-xylenol (IV), m. 90-110°. I (50 g.) in 600 cc. AcOH with 103 g. Br in 200 cc. AcOH (3 hrs.) gives 87.5% 1,3,4,6,2-Me2Br2ClC6OH, m. 164° (cf. Lesser and Gad, C.A. 17, 3181). IV with an addnl, mol. of Cl gives a nearly quant. yield of 2,6-dichloro-4-bromo-3,5-xylenol, m. 182°; in larger quantities, it was prepared in 87% yield by slowly adding 37 g. Cl in 500 cc. AcOH to 52 g. III in 500 cc. cold AcOH. II (15 g.) in 120 cc. AcOH, treated (below 20°) with 4.5 g. Cl in 150 cc. AcOH, gives 97.6% 4,6-dichloro-2-bromo-3,5-xylenol (V), m. 165-6°; V results in 84.7% yield by slowly adding 10 g. Br in 80 cc. AcOH to 12 g. 1,3,2,4,5-Me2Cl2C6HOH in 120 cc. AcOH at room temperature Oxidation of V at 100° with HNO3 (d. 1.42) gives 5-chloro-3-bromo-m-xyloquinone (VI), yellow, m. 170-1°. IV (14.7 g.) and 10 g. Br in AcOH (temperature below 20°) give 90% 6-chloro-2,4-dibromo-3,5-xylenol, m. 170°; oxidation gives VI. 1,3,5,2-Me2ClC6H2OH (VII) (15 g.) in 150 cc. CHCl3 containing a little AlBr3, treated rapidly with 16.8 g. Br in 75 cc. CHCl3 and the mixture kept 2 hrs. at 50°, give 96.6% 4-chloro-3-bromo-2,6-xylenol (VIII), m. 86-7°. Molten VIII (2.3 g.), added (15 min.) to 7.4 g. liquid Br at room temperature, gives 96.9% 4-chloro-3,5-dibromo-2,6-

xylenol (IX), m. 188-9°; addition of 2 mols. Br in 12 times its weight of H2O to finely ground IX at room temperature gives 3,5-dibromo-m-xyloquinone (X) and a scarlet adduct of 1 mol. X and 2 mols. IX, m. 141-2° (also prepared from the 2 components). IX is prepared in 98.5% yield by gradual addition of 1 mol. VII to 4 mols. liquid Br (1 hr.) at 20°. Addition of 5.75g. molten 1,3,4,5,2-Me2Cl2C6HOH to 19.3 g. Br at room temperature gives 98.4% 4,5-dichloro-3-bromo-2,6-xylenol, pale yellow, m. 188°. Improved directions are given for the preparation of the 4-Br, 3,4-di-Br, and 3,4,5-tri-Br derivs. of 2,6-Me2C6H3OH.

IT **408314-44-3**, 3,5-Xylenol, 4-bromo-2,6-dichloro-
408326-71-6, 2,6-Xylenol, 3,5-dibromo-4-chloro-
857974-71-1, m-Xyloquinone, 3,5-dibromo-, compound with 3,5-dibromo-4-chloro-2,6-xylenol **859784-53-5**, 3,5-Xylenol, 2,4-dibromo-6-chloro- **874519-00-3**, 3,5-Xylenol, 2-bromo-4,6-dichloro-
(preparation of)

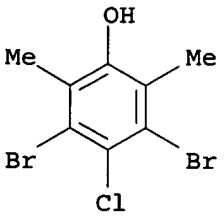
RN 408314-44-3 CAPLUS

CN Phenol, 4-bromo-2,6-dichloro-3,5-dimethyl- (9CI) (CA INDEX NAME)



RN 408326-71-6 CAPLUS

CN Phenol, 3,5-dibromo-4-chloro-2,6-dimethyl- (9CI) (CA INDEX NAME)

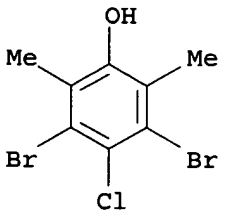


RN 857974-71-1 CAPLUS

CN m-Xyloquinone, 3,5-dibromo-, compd. with 3,5-dibromo-4-chloro-2,6-xylenol (5CI) (CA INDEX NAME)

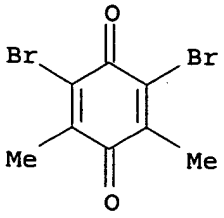
CM 1

CRN 408326-71-6
CMF C8 H7 Br2 Cl O

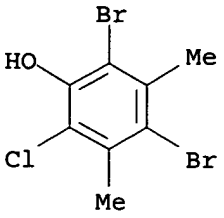


CM 2

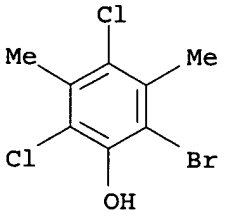
CRN 87405-27-4
CMF C8 H6 Br2 O2



RN 859784-53-5 CAPLUS
CN 3,5-Xylenol, 2,4-dibromo-6-chloro- (5CI) (CA INDEX NAME)



RN 874519-00-3 CAPLUS
CN 3,5-Xylenol, 2-bromo-4,6-dichloro- (5CI) (CA INDEX NAME)

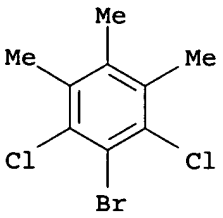


L11 ANSWER 11 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1946:4859 CAPLUS
DOCUMENT NUMBER: 40:4859
ORIGINAL REFERENCE NO.: 40:782h-i,783a-c
TITLE: Melting points and unit cells of the methylbenzenes
AUTHOR(S): Beacall, T.
SOURCE: Transactions of the Faraday Society (**1945**), 41, 472-9
CODEN: TFSOA4; ISSN: 0014-7672
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB cf. C.A. 38, 671.6. M.ps. of the 12 possible Me derivs. of benzene and of the following 30 halogen derivs. are related to the dimensions of the unit cells as determined from available x-ray analyses: pentabromotoluene, pentachlorotoluene, 6-chloro-2,3,4,5-tetrabromotoluene, 2,3,5,6-tetrachloro-4-bromotoluene, tetrabromo derivs. of o-, m-, and p-xylenes, tetrachloro derivs. of o-, m-, and p-xylenes, 6-chloro-2,3,5-tribromo-p-xylene, 3,6-dichloro-2,5-dibromo-p-xylene, 3,5,6-trichloro-2-bromo-p-xylene, 4,6-dichloro-3,5-dibromo-o-xylene, 4,6-dichloro-2,5-dibromo-m-xylene, 2,6-dichloro-4,5-dibromo-m-xylene, 2,4,6-tribromo-1,3,5-trimethylbenzene, 3,5,6-tribromo-1,2,4-trimethylbenzene, 4,5,6-tribromo-1,2,3-trimethylbenzene, 2,4,6-trichloro-1,3,5-trimethylbenzene, 3,5,6-trichloro-1,2,4-trimethylbenzene, 4,5,6-trichloro-1,2,3-trimethylbenzene, 4,6-dichloro-5-bromo-1,2,3-trimethylbenzene, 3,6-dibromo-1,2,4,5-tetramethylbenzene, 4,6-dibromo-1,2,3,5-tetramethylbenzene, 5,6-dibromo-1,2,3,4-tetramethylbenzene, 3,6-dichloro-1,2,4,5-tetramethylbenzene, 5,6-dichloro-1,2,3,4-tetramethylbenzene,

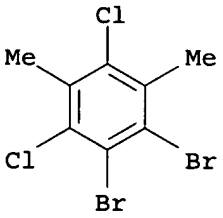
bromopentamethylbenzene, chloropentamethylbenzene. The m.ps. of p-xylene, durene, and hexamethylbenzene form a geometrical series; the vols. of the unit cell per mol. approx. to an arithmetical series. It is suggested that in these mols. the pairs of Me groups in para position are linked to Me groups of adjacent mols. The volume of the unit cell of pentabromotoluene as determined from its mol. weight and d. is in agreement with that of benzene plus increments due to the 3 pairs of substituents. The lowering in m.p. by introduction of a single Me group points to a head-to-tail linkage in the unit cell. A tentative structure for the unit cell of mesitylene is suggested after the low m.p. of mesitylene is contrasted with the comparatively high m.p. of the sym. trihalobenzenes.

IT **679427-57-7**, Hemimellitene, 5-bromo-4,6-dichloro-
679428-36-5, m-Xylene, 4,5-dibromo-2,6-dichloro-
679428-38-7, m-Xylene, 2,5-dibromo-4,6-dichloro-
679428-39-8, p-Xylene, 2,3,5-tribromo-6-chloro-
679428-40-1, o-Xylene, 3,5-dibromo-4,6-dichloro-
679428-41-2, p-Xylene, 2-bromo-3,5,6-trichloro-
(m.p. and unit cell of)

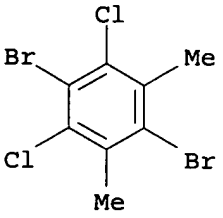
RN 679427-57-7 CAPLUS
CN Hemimellitene, 5-bromo-4,6-dichloro- (4CI) (CA INDEX NAME)



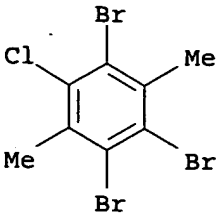
RN 679428-36-5 CAPLUS
CN m-Xylene, 4,5-dibromo-2,6-dichloro- (4CI) (CA INDEX NAME)



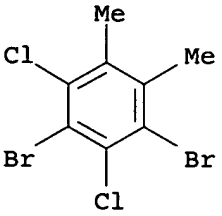
RN 679428-38-7 CAPLUS
CN m-Xylene, 2,5-dibromo-4,6-dichloro- (4CI) (CA INDEX NAME)



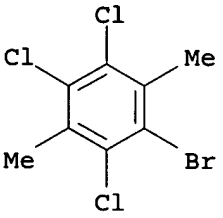
RN 679428-39-8 CAPLUS
CN p-Xylene, 2,3,5-tribromo-6-chloro- (4CI) (CA INDEX NAME)



RN 679428-40-1 CAPLUS
CN o-Xylene, 3,5-dibromo-4,6-dichloro- (4CI) (CA INDEX NAME)



RN 679428-41-2 CAPLUS
CN p-Xylene, 2-bromo-3,5,6-trichloro- (4CI) (CA INDEX NAME)



L11 ANSWER 12 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1945:29888 CAPLUS
DOCUMENT NUMBER: 39:29888
ORIGINAL REFERENCE NO.: 39:4855i,4856a-i
TITLE: Synthesis and antitubercular studies of halogenated phenyl ethers
AUTHOR(S): Burger, Alfred; Wilson, Elizabeth L.; Brindley, C. O.; Bernheim, Frederick
SOURCE: Journal of the American Chemical Society (**1945**), 67, 1416-19
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

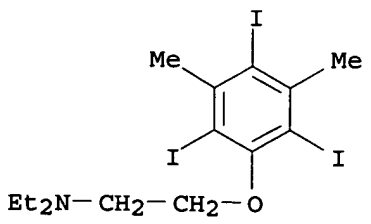
AB In general, 100 g. of the phenol in a mixture of 800 cc. 20% NH4OH and 200 to 800 cc. MeOH was treated with the calculated amount of iodine solution (containing 1 part of iodine and 2 parts KI in 4 parts of H2O) at such a rate that the brown color never persisted for any length of time; the crude yields were above 90% but 5-20% was lost during purification; the anisoles were prepared from the Na phenolates and Me2SO4 and the lauryl ethers from the Na salts and lauryl iodide in MeOH. 2,6-Diiodo-4-chloroanisole, m. 65°; 2,4,6-triiodo-1-dodecoxybenzene (I), m. 63-4°; 3-Me derivative (II), m. 47-8°; 2,4,6-triiodo-3,5-dimethylphenol, m. 175-7° (decomposition); 2,4-diiodo-4-tert-amylphenol, m. 62°; 2-bromo-4-phenyl-6-iodophenol, m. 86.5-8°; 2,4-diiodo-4-tert-butylphenol, m. 82°. Most of the basic ethers were prepared from 0.1 mole of the phenol in 0.2 mole of Na in MeOH at 30-50° and 0.12 mole of a dialkylaminoalkyl chloride-HCl on refluxing 5 to 12 hrs.; 0.1 mole of NaI was added as a catalyst; the yields averaged 25 to 35%; with Me2N(CH2)2Cl the yield was sometimes below 5%; dialkylaminopropyl chlorides gave from 50 to 90% yields. Ethers of 2,4,6-I3C6H2OH (HCl salts

in all cases unless stated): 2-dimethylaminoethyl, m. 226° (decomposition); 2-dibutylaminoethyl, m. 192-4° (decomposition); 2-morpholinoethyl, m. 240-2° (decomposition) (free base, m. 130-1°); 3-diethylaminopropyl, m. 190-2° (decomposition). 2,6,4-I2BrC6H2OH: 2-diethylaminoethyl, m. 174-7° (decomposition). 2,6,4-I2ClC6H2OH: 2-diethylaminoethyl, m. 182° (decomposition); 3-diethylaminopropyl, m. 214° (decomposition). 2,4,6-I2ClC6H2OH: diethylaminoethyl, m. 155-6°; 3-(2-methylpiperidino)propyl, m. 188° (decomposition). 2,6,4-I2PhC6H2OH:2-diethylaminoethyl, m. 198-9° (decomposition). 2,6,4-I2MeC6H2OH: 2-diethylaminoethyl, m. 166.5°. 2,4,6-I2MeC6H2OH: 2-diethylaminoethyl, m. 151-2°. 2,4,6,3-I3MeC6HOH: 2-diethylaminoethyl, m. 173-4°; 2-dibutylaminoethyl, m. 190-3° (decomposition). 2,4,6,3,5-I3Me2C6OH: 2-diethylaminoethyl, m. 209° (decomposition). 2,6,4-IBrPhC6H2OH: 2-diethylaminoethyl, m. 190-1° (decomposition). 2,6-Diiodo-4-phenylazophenol: 2-diethylaminoethyl, m. 188-90° (decomposition). 2,4,6-Br3C6H2OH: 2-dimethylaminoethyl, m. 194°; 2-diethylaminoethyl, m. 149°. 2,4,6-Cl3C6H2OH: 2-dimethylaminoethyl (free base), b4 131-3° (picrate, m. 192-3°); 2-diethylaminoethyl, m. 160-2°; 3-(2-methylpiperidino)propyl, m. 156-7°. 2,4,5-Cl3C6H2OH: diethylaminomethyl, m. 157-9° (decomposition); 2-dimethylaminoethyl, m. 210-11°; 2-diethylaminoethyl, m. 183°; 2-dibutylaminoethyl, m. 124°; 3-diethylaminopropyl, m. 179°; 3-dibutylaminopropyl (free base), b2 180-90° (picrate, m. 119-20°); 3-(2-methylpiperidino)propyl, m. 229-30°; 2-butylaminoethyl, m. 122°. 2,4-Cl2C6H3OH: 2-dibutylaminoethyl, m. 295-7°. Cl5C6OH: 3-(2-methylpiperidino)propyl, m. 224° (decomposition). PhOH: 2-diethylaminoethyl, m. 137.5°. 4-tert-BuC6H4OH: 2-diethylaminoethyl, m. 158-60°. 4-tert-AmC6H4OH: 2-diethylaminoethyl, m. 128-31°. 3-Me2C6H4OH: 2-dibutylaminoethyl (free base), b1 147-50°. 3,5-Me2C6H3OH: 2-diethylaminoethyl, m. 132°. In general, chemotherapeutic activity was not restricted to compds. containing aliphatic NH2 groups; the ethers containing the 3-(2-methylpiperidino)propyl radical were highly active. Activity, but also toxicity in guinea pigs, was usually greater in dialkylaminopropyl than in the corresponding dialkylaminoethyl ethers; shortening of the ethylene group, in a trihalophenoxy(diethylamino)methane derivative, caused complete loss of antitubercular action. Replacement of the Bu2N group in an active ether by the BuNH group also abolished activity. Replacement of nuclear iodine by other halogens did not lead to a definite pattern correlating chemical structure to antitubercular activity. Nuclear Br had a slightly dystherapeutic effect, whereas several of the poly-Cl derivs. rivaled analogous iodinated compds. Nuclear iodine does not appear superior to other halogens in the tuberculostatic activity of this series. I and II has no activity, probably because of the insoly. of these ethers in polar solvents.

IT **854640-87-2**, Triethylamine, 2-(2,4,6-triiodo-3,5-xylyloxy)-, hydrochloride
(preparation of)

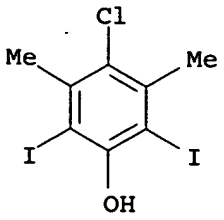
RN 854640-87-2 CAPLUS

CN Triethylamine, 2-(2,4,6-triiodo-3,5-xylyloxy)-, hydrochloride (4CI) (CA INDEX NAME)

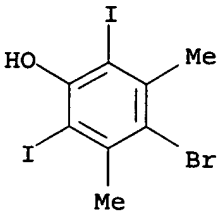


● HCl

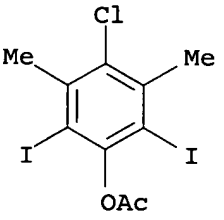
L11 ANSWER 13 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1938:41756 CAPLUS
DOCUMENT NUMBER: 32:41756
ORIGINAL REFERENCE NO.: 32:5802h-i,5803a-f
TITLE: Aristols. Preparation and constitution of aristols derived from p-xylenol and sym-m-xylenol. II
AUTHOR(S): Bordeianu, C. V.
SOURCE: Annales Scientifiques de l'Universite de Jassy (1937), 23(Pt. I), 240-64
CODEN: ASUJAH; ISSN: 0365-7264
DOCUMENT TYPE: Journal
LANGUAGE: French
GI For diagram(s), see printed CA Issue.
AB By direct mercuration of the corresponding halogen-substituted xylenol (cf. preceding abstrs.) the following compds. were prepared: 2,5-dimethyl-4-bromo-6-acetoxymercuriphenol (I), 91% yield; 2,5-dimethyl-4-iodo-6-acetoxymercuriphenol (II), 80-90% yield; 3,5-dimethyl-4-bromo-6-acetoxymercuriphenol (III), almost quant. yield; 3,5-dimethyl-4-chloro-6-acetoxymercuriphenol (IV); and 3,5-dimethyl-4-chloro-5-iodo-2-acetoxymercuriphenol (V). All were insol. in H2O, soluble in glacial AcOH and soluble without decomposition in NaOH. Treated with CO2 in the presence of alkali, they gave the internal Hg oxides (cf. Bordeianu, loc. cit.) and with NaI and I (Dimroth's reaction) gave iodides in which the I replaced the AcOHg group. Thus, I gave 90% of 2,5-dimethyl-4-bromo-6-iodophenol (VI), m. 64-5° (from petr. ether); acetate, m. 73° (from glacial AcOH). II yielded 2,5-dimethyl-4,6-diiodophenol (VII), m. 63° (from glacial AcOH), identical with the compound previously reported (C. A. 30, 1760.5) which is oxidized by K2Cr2O7 in AcOH to give 60% of a quinone (from analogy with iodothymoquinone, presumably of the structure CO.CMe:CI.CO.CMe:CH), m. 79-80° (from 60-70% alc.), deep orange, sublimes and steam-distils (monooxime yellow, insol. in H2O, decomps. 170°; dioxime could not be prepared). III yielded 3,5-dimethyl-4-bromo-6-iodophenol (VIII), m. 85.5° (from petr. ether). IV gave 3,5-dimethyl-4-chloro-6-iodophenol (IX), colorless, m. 92-3° (from petr. ether); acetate, m. 86° (from alc.). V gave 100% of 3,5-dimethyl-2,6-diiodo-4-chlorophenol (X), colorless, m. 131-2° (from alc., Et2O or AcOH), which can be prepared also from 3,5-dimethyl-4-chlorophenol with I and alkali (poor yields) or with I and NH3 in MeOH; acetate, m. 147-8° (from glacial AcOH). Compds. VI-X are colorless and soluble in dilute alkalies. Oxidizing agents in alkaline media convert the dihalides to aristols, insol. in alkalies; e.g., VI and VII with K2S2O8 and KOH or with I and KOH give quant. yields of the aristol 5,5'-diiodo-3,6,3',6'-dimethyl-4,4'-biphenoquinone (CH:CMe.CO.CI:CMe.C:)2, also made by the action of alkali on p-xylenol. Similarly the aristol 3,5,3',5'-tetraiodo-2,6,2',6'-tetramethyl-4,4'-biphenoquinone (XI) is prepared in quant. yields from X, from 2,3,5-triiodo-3,5-dimethylphenol (XII) and from 2,6-diiodo-4-bromo-3,5-dimethylphenol (XIII). The reasons for assigning the particular structures are similar to those set forth in C. A. 28, 2339.1, and the fact that Br from XIII is recovered quant. as the bromate. The mol.-weight determination on XI gave values twice as great as required by the formula assigned, so the substance must actually exist as a dimer. B. claims that while the preparation of XII was described in the article abstracted in C. A. 30, 1760.5, it was called (incorrectly) the diiodide-the data on mol. weight and % I were for the diiodide, but the properties and preparation were for the triiodide (XII), whose purification is described in detail in the present article. XIII is synthesized directly from I and 4-bromo-3,5-dimethylphenol, only if 5 mols. of NaOH and 4 mols. of I are used for each mol. of XIV. If less I is employed, XII is precipitated The aristol which is mentioned as being formed from IX is not described.
IT **791626-94-3**, 3,5-Xylenol, 4-chloro-2,6-diiodo- **854672-30-3**, 3,5-Xylenol, 4-bromo-2,6-diiodo- **856351-96-7**, 3,5-Xylenol, 4-chloro-2,6-diiodo-, acetate
(preparation of)
RN 791626-94-3 CAPLUS
CN 3,5-Xylenol, 4-chloro-2,6-diiodo- (5CI) (CA INDEX NAME)



RN 854672-30-3 CAPLUS
CN 3,5-Xylenol, 4-bromo-2,6-diiodo- (4CI) (CA INDEX NAME)



RN 856351-96-7 CAPLUS
CN 3,5-Xylenol, 4-chloro-2,6-diiodo-, acetate (4CI) (CA INDEX NAME)

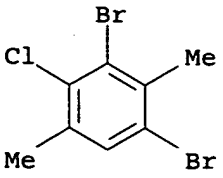


L11 ANSWER 14 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1937:56695 CAPLUS
DOCUMENT NUMBER: 31:56695
ORIGINAL REFERENCE NO.: 31:7856i,7857a-c
TITLE: 3,5-Dibromo-p-xylidine; 3,5,6-tribromo-p-xylidine and some of their derivatives
AUTHOR(S): Bures, E.; Meskan, F.
SOURCE: Casopis Ceskoslovenskeho Lekarnictva (**1937**), 17, 149-60
CODEN: CCLEA3
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

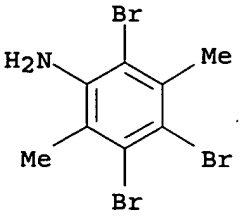
AB The bromination of an alc. solution of p-xylidine, at room temperature and without an access to sunlight, resulted in the formation of 3,5-dibromo-p-xylidine. The constitution of this compound was proved by its transformation into 2,3,5-tribromo-p-xylenol. From 3,5-dibromo-p-xylidine there were prepared the following derivs.: 3,5-dibromo-p-acetxylidine, b. 163°; 3,5-dibromo-p-diacetxylidine, b. 56°; 3,5-dibromo-p-benzoylxylidine, b. 192°; 3,5-dibromo-p-xylene, b. 36°; 3,5-dibromo-2-chloro-p-xylene, b. 85°; 2,3,5-tribromo-p-xylene, b. 89°; 1,4-dimethyl-3,5-di-bromo-2-benzonitrile, b. 97°, and 3,5-dibromo-p-xylenol, b. 82°. From this last compound there were prepared the Hg and Bi xylenolates and its Me ether. By the careful bromination of the diluted solution of p-acetxylidine in glacial AcOH, there was obtained 3,5,6-tribromo-1,4-dimethyl-2-acetaminobenzene.

IT **854662-93-4**, p-Xylene, 3,5-dibromo-2-chloro- **854673-69-1**, 2,5-Xylidine, 3,4,6-tribromo- **856356-94-0**, 2,5-Acetoxylide, 3,4,6-tribromo-
(preparation of)

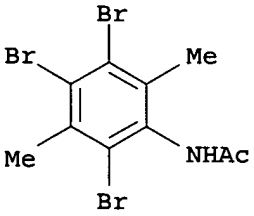
RN 854662-93-4 CAPLUS
CN p-Xylene, 3,5-dibromo-2-chloro- (4CI) (CA INDEX NAME)



RN 854673-69-1 CAPLUS
CN 2,5-Xylidine, 3,4,6-tribromo- (4CI) (CA INDEX NAME)



RN 856356-94-0 CAPLUS
CN 2,5-Acetoxylide, 3,4,6-tribromo- (4CI) (CA INDEX NAME)



L11 ANSWER 15 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1936:13394 CAPLUS
DOCUMENT NUMBER: 30:13394
ORIGINAL REFERENCE NO.: 30:1751i,1752a-g
TITLE: Polymethylenes. XV. The Jacobsen reaction 4
AUTHOR(S): Smith, Lee I.; Moyle, Clarence L.
SOURCE: Journal of the American Chemical Society (**1936**), 58, 1-10
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

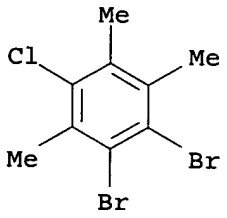
AB Chlorodurene, chloroisodurene and chloroprehnitene rearrange in contact with H_2SO_4 to give chloropentamethyl-benzene (I) and 3-chloropseudocumene-5-sulfonic acid (II), a Me group migrating in such a manner that the same chlorotrimethylbenzene derivative resulted. Chloroisodurene also gives a small quantity of a compound, $C_{20}H_{24}Cl_2$, m. 209.5°, the structure of which is not known. 5-Chloropseudocumene and the 6-isomer give II; chloromesitylene and 4-chlorohemimellitene were stable toward H_2SO_4. Bromomesitylene gives mesitylenesulfonic acid and di- or tribromomesitylene, depending upon the temperature; 5-bromopseudocumene gives largely 3-bromopseudo-cumene-5-sulfonic acid, together with a small quantity of tribromopseudocumene; no pseudocumene-5-sulfonic acid was found. The following did not rearrange with H_2SO_4: hemimellitene, 5-nitropseudocumene, 5-pseudocumidine, Me pentamethylbenzenesulfonate, pentamethyl-cyclohexane, 2,3-$C_{10}H_6Me_2$ and p-BrC_6H_4Ph. Yields are given, together with the conditions used. I was identified by analysis and conversion into C_6HMe_5; II was identified by conversion into the amide, m. 182°. 3-Chloro-5,6-dinitropseudocumene is reduced by $SnCl_2$ in

EtOH-HCl to the di-NH2 derivative, m. 136.5°, stable in the air for some time; phenanthrenequinone (III) in AcOH-EtOH gives 12-chloro-10,11,13-trimethylphenanthrophenazine, yellow, m. 330.5-1°. Reduction of the di-NO2 compound with SnCl2 in AcOH-HCl yields 6-chloro-2,4,5,7-tetramethyl-benzimidazole, m. 250-1°. 5-Chloro-1,3-diaminomesitylene, m. 137-8°; this does not react with III or form a benzimidazole. Nitration of Na 4-chlorohemimellitene-sulfonate gives 4-chloro-5,6-dinitrohemimellitene, m. 182-2.5°; SnCl2 in AcOH yields 7-chloro-2,4,5,6-tetramethyl-benzimidazole, m. 286.5-7.5°. 10,11,13-Trimethylphenanthrophenazine, yellow, m. 253°. Many other details are given of the preparation of products used for the identification of the compds. formed in the rearrangement, as well as starting materials. Na chloromesitylene-sulfonate crystallizes with 0.5 mol. H2O, as does the Br derivative; bromomesitylene-sulfonamide, m. 160-60.5°. 3-Chloro-5,6-dibromopseudocumene, m. 224°; 6-chloro-3,5-dinitro-pseudocumene, m. 162°. 6-Bromo-5-pseudocumidine, m. 69° (56.8% yield). 4-Chloro-5,6-dibromohemimellitene, m. 229-30°; 13-chloro-10,11,12-trimethylphenanthrophenazine, yellow, m. 346.5-7°. 7-Chloro-2,4,5,6-tetramethyl-benzimidazole, m. 288.5°. 10,11,12-Trimethylphenanthrophenazine, orange, m. 311°. Pentamethylbenzene-methanesulfonate, m. 91-1.5°. The ease of migration of groups present in the chloro- and bromotetramethyl-benzenes is in the order Br > Me > Cl; in case of the corresponding derivs. of the C6H3Me3 the order is Br > Cl > Me. Attempts to find mild conditions which would cause Jacobsen rearrangements without producing amorphous by-products were unsuccessful. Dilution of the H2SO4 with 10% of H2O or with H3PO4 or AcOH inhibited the rearrangement and the side reaction as well. The use of CaCl2, Mg(ClO4)2, PhSO3H, AcOH or H3PO4 merely caused hydrolysis of the sulfonic acid to the hydrocarbon and no reagents or conditions were found, other than those already known, which would cause any rearrangements to take place. Little can be said with regard to the mechanism of the reaction.

IT **856070-13-8**, Pseudocumene, 5,6-dibromo-3-chloro-
859774-87-1, Hemimellitene, 4,5-dibromo-6-chloro-
(preparation of)

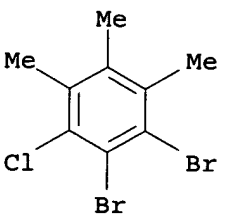
RN 856070-13-8 CAPLUS

CN Pseudocumene, 5,6-dibromo-3-chloro- (3CI) (CA INDEX NAME)



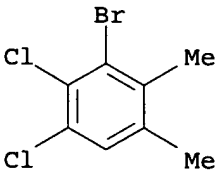
RN 859774-87-1 CAPLUS

CN Hemimellitene, 4,5-dibromo-6-chloro- (3CI) (CA INDEX NAME)



L11 ANSWER 16 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1935:10887 CAPLUS
DOCUMENT NUMBER: 29:10887
ORIGINAL REFERENCE NO.: 29:1398e-g

TITLE: Dichloro-o-xylene. II
AUTHOR(S): Hinkel, Leonard E.; Ayling, Ernest E.; Walters, Thomas M.
SOURCE: Journal of the Chemical Society (1934) 1946-8
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB cf. C. A. 22, 3638. 6-Chloro-o-3-xylidine through the Sandmeyer reaction gives 70% of 3,6-dichloro-o-xylene (I), b760 234° m. 29°; chlorination gives the 3,4,5,6-tetra-Cl derivative; nitration gives the 4-NO2 derivative (II), m. 84°; reduction with Fe and 5% AcOH gives 96% of 3,6-dichloro-o-4-xylidine, m. 54° (Ac derivative, m. 146°). The 4,5-di-NO2 derivative of I m. 174°; the 4,5-di-NH2 derivative m. 176° (phenazine derivative, m. above 250°). The 4,5-di-Br derivative of I m. 238°. 6-Chloro-4-nitro-o-3-xylidine through the Sandmeyer reaction gives II. 5-Chloro-o-4-xylidine gives 71% of 4,5-dichloro-o-xylene, m. 76°; 3,6-di-Br derivative, m. 232°; the 3-Br derivative, from 4,5-dichloro-o-3-xylidine, m. 111°. Chlorination of 3-chloro-o-xylene gives the 3,4-di-Cl derivative only; the 4-Cl derivative gives only the 4,5-di-Cl derivative Nitration of 4-chloro-o-xylene gives the 5-NO2 derivative, m. 63°, and a di-NO2 derivative, m. 111°.
IT **854861-53-3**, o-Xylene, 3-bromo-4,5-dichloro- (preparation of)
RN 854861-53-3 CAPLUS
CN o-Xylene, 3-bromo-4,5-dichloro- (3CI) (CA INDEX NAME)



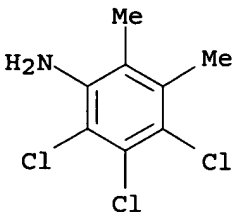
L11 ANSWER 17 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1934:28468 CAPLUS
DOCUMENT NUMBER: 28:28468
ORIGINAL REFERENCE NO.: 28:3389i,3390a-e
TITLE: Chlorination of the aceto-o-xylides
AUTHOR(S): Hinkel, Leonard E.; Ayling, Ernest E.; Walters, Thomas M.
SOURCE: Journal of the Chemical Society (1934) 283-7
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB The extent of chlorination of aceto-o-3-xylide (I) depends mainly on the temperature; the yield of Cl derivs. varied with the strength of the AcOH used and was greatest (65%) in glacial AcOH at 10-12°; with dichloramine T the 4-Cl (II) and the 6-Cl derivative (III) were obtained in 17 and 65% yields, resp. II m. 169° and is difficult of isolation because of its ready solubility in organic solvents. With 2 mols. Cl in glacial AcOH above 40° I gives 4,6-dichloroaceto-o-3-xylide (IV), m. 188°; hydrolysis gives 4,6-dichloro-o-3-xylidine, m. 44°; at lower temps. some III was also formed; with excess Cl and an increase of temperature a mixture of IV and the 4,5,6-tri-Cl derivative was formed, which on hydrolysis gives 4,5,6-trichloro-o-3-xylidine, m. 207°. Chlorination of III in AcOH at 50° gives IV. III and HNO3 in AcOH, heated on a steam bath for 30 min., give 6-chloro-4-nitroaceto-o-3-xylide, m. 196.5°; hydrolysis with 40% H2SO4 gives 6-chloro-4-nitro-o-3-xylidine, m. 143°; reduction gives an o-diamine. I and KOCl give N-chloroaceto-o-3-xylide, m. 94.5°, readily transformed into a mixture of II and III. III yields a N,6-di-Cl derivative, m. 81°, transformed into IV. IV yields a N,4,6-tri-Cl derivative, isomerization of which regenerates IV. Chlorination of aceto-o-4-xylide (V) in AcOH with 1 mol. of Cl or with dichloramine T gives a mixture of the 5-Cl derivative (VI)

and the 3-Cl derivative (VII), m. 114°; hydrolysis of the latter gives 3-chloro-o-4-xylidine, m. 26°; both VI and VII are much less basic than II and III, their salts with HCl and H2SO4 being completely dissociated in dilute solution The yield of VI and VII from V and dichloramine T in CHCl3 are 48 and 19%, resp., 22% V being recovered. N-Chloroaceto-o-4-xylide, m. 55°; warming in CHCl3 or AcOH gives 50% of VI and VII and 25% V. The N,5-di-Cl derivative m. 74° (90% yield); it is slowly transformed into the 3,5-di-Cl derivative in CHCl3 or AcOH at room temperature; warming in AcOH containing a little H2SO4 for 2 days gives 48% of the 3,5-di-Cl derivative and 10% of the 5-Cl derivative 3-Nitro-o-4-xylidine through the diazo reaction gives 60% of 4-chloro-3-nitro-o-xylene, pale yellow, m. 75°; reduction and acetylation gives II. The structure of IV follows from the removal of the NH2 groups from the free base, yielding 3,5-dichloro-o-xylene. 5-Chloro-4-nitro-o-xylene, pale yellow, m. 63°; reduction and acetylation gives VI; VII was synthesized from 4-nitro-o-3-xylidine.

IT **854857-43-5**, 2,3-Xylidine, 4,5,6-trichloro-
(preparation of)

RN 854857-43-5 CAPLUS

CN 2,3-Xylidine, 4,5,6-trichloro- (3CI) (CA INDEX NAME)



L11 ANSWER 18 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1930:53093 CAPLUS

DOCUMENT NUMBER: 24:53093

ORIGINAL REFERENCE NO.: 24:5731i,5732a-b

TITLE: The nitration of 5-bromo-1,3-dimethyl-4-acetamidobenzene and 3,5 -dibromo-1,4-dimethyl-2-acetamidobenzene and some derivatives

AUTHOR(S): Bures, E.; Smetana, J.

SOURCE: Casopis Ceskoslovenskeho Lekarnictva (**1930**), 10, 99-102,131-7,160-4
CODEN: CCLEA3

DOCUMENT TYPE: Journal

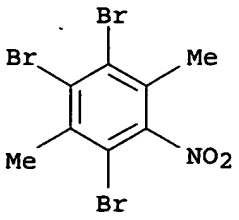
LANGUAGE: Unavailable

AB 6,2,4-BrMe2C6H2NH2, m. 49-50°, wits converted into its Ac derivative m. 197-8°, which on nitration gave 5-bromo-2,6-dinitro-1,3-dimethyl-4-acetamidobenzene, m. 278°, from which were prepared: 5-bromo-2,6-dinitro-1,3-dimethyl-4-aminobenzen, m. 171-2°; 5-bromo-2,6-dinitro 1,3-dimethylbenzene, m. 67°; 4,5-dibromo-2,6-dinitro-1,3-dimethylbenzene, m. 193°. From p-xylidinc were prepared 3,5-dibromo-1,4-dimethyl-2-aminobenzene, m. 65°. and its Ac derivative, m. 193°. Nitration of 2,5,4,6-Me2Br2C5HNHAc gave 3,5 dibromo-6-nitro-1,4-dimethyl-2-acetamidobenzene, m. 264-.5 °, from which were prepared: 3,5-dibromo-6-nitro-1,4-dimethyl-2-aminobenzene, m. 176°; 3,5-dibromo-6-nirtro-1,4 dimethylbenzene, m. 69°; 2,3,5-tribromo-6-nitro-1,4-dimethylbenzene, m. 207°; 3,5. dibromo-2-iodo-6-nitro-1,4-dimethylbenzene, m. 204°.

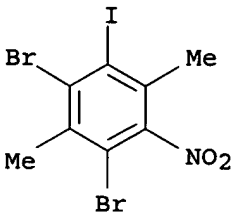
IT **854861-22-6**, p-Xylene, 2,3,5-tribromo-6-nitro- **854861-35-1**, p-Xylene, 2,6-dibromo-3-iodo-5-nitro-
(preparation of)

RN 854861-22-6 CAPLUS

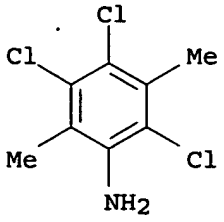
CN p-Xylene, 2,3,5-tribromo-6-nitro- (3CI) (CA INDEX NAME)



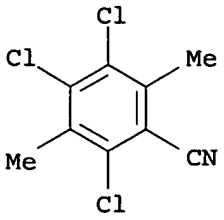
RN 854861-35-1 CAPLUS
CN p-Xylene, 2,6-dibromo-3-iodo-5-nitro- (3CI) (CA INDEX NAME)



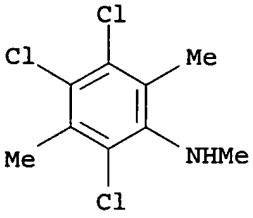
L11 ANSWER 19 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1930:16898 CAPLUS
DOCUMENT NUMBER: 24:16898
ORIGINAL REFERENCE NO.: 24:1851b-d
TITLE: 1,4-Dimethyl-3,5,6-trichloro-2-aminobenzene and some of its derivatives
AUTHOR(S): Bures, E.; Rubes, T.
SOURCE: Collection of Czechoslovak Chemical Communications (**1929**), 1, 648-57
CODEN: CCCCAK; ISSN: 0010-0765
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB 1,4-Dimethyl-2-acetamidobenzene in a large excess of anhydrous AcOH gives quantitatively 1,4-dimethyl-3,5,6-trichloro-2-acetamidobenzene (I), m. 222°, by direct chlorination. Saponification of I gives quant. the 2-amino compound (II), m. 206°, whose Bz derivative m. 223°. II and Me2SO4 give the 2-methylamino derivative, m. 62°. The picrate of II, red-purple crystals, m. above 100° (decomposition) and is decomposed into its constituents by H2O. By diazotization of II, 1,4-dimethyl-3,5,6-trichloro-2- hydroxybenzene (III), m. 175°, is obtained: its alkaline solution treated with Me2SO4 gives the corresponding MeO derivative, m. 91°, while Et2SO4 gives the EtO derivative, m. 79°. The benzoate of III m. 101°, and the acelate m. 103°. A solution of the Na salt of III, treated with HgCl2, ppts. a basic Hg salt containing 28.22% Hg. A basic Bi salt can be similarly obtained. 1,4-Dimethyl-3,5,6-trichlorobenzene, m. 96°, is obtained from II by diazotization; 1,4-dimethyl-2,3,5,6-tetrachlorobenzene, m. 223°, is similarly obtained from II; 1,4-dimethyl-3,5,6-trichloro-2-cyanobenzene, m. 213°, is also similarly obtained.
IT **857974-28-8**, 2,5-Xylidine, 3,4,6-trichloro- **859202-54-3**, Isoxylonitrile, 3,4,6-trichloro- **872275-60-0**, 2,5-Xylidine, 3,4,6-trichloro-N-methyl- **873979-22-7**, 2,5-Acetoxylide, 3,4,6-trichloro- **873987-55-4**, 2,5-Benzoxylide, 3',4',6'-trichloro- **876486-92-9**, Phenetole, 2,4,5-trichloro-3,6-dimethyl-
(preparation of)
RN 857974-28-8 CAPLUS
CN 2,5-Xylidine, 3,4,6-trichloro- (5CI) (CA INDEX NAME)



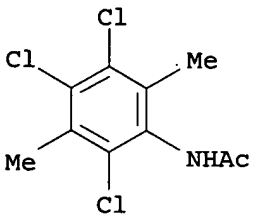
RN 859202-54-3 CAPLUS
CN 2,5-Xylonitrile, 3,4,6-trichloro- (3CI) (CA INDEX NAME)



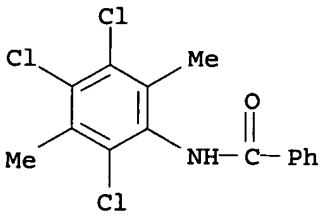
RN 872275-60-0 CAPLUS
CN 2,5-Xylidine, 3,4,6-trichloro-N-methyl- (3CI) (CA INDEX NAME)



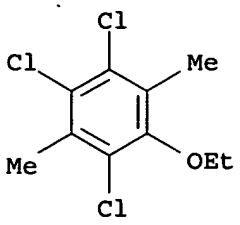
RN 873979-22-7 CAPLUS
CN 2,5-Acetoxylide, 3,4,6-trichloro- (3CI) (CA INDEX NAME)



RN 873987-55-4 CAPLUS
CN 2,5-Benzoxylide, 3',4',6'-trichloro- (3CI) (CA INDEX NAME)



RN 876486-92-9 CAPLUS
CN INDEX NAME NOT YET ASSIGNED



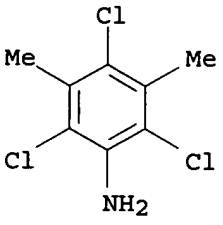
L11 ANSWER 20 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1930:6485 CAPLUS
DOCUMENT NUMBER: 24:6485
ORIGINAL REFERENCE NO.: 24:729i,730a-b
TITLE: Azo dyes
PATENT ASSIGNEE(S): I. G. Farbenindustrie AG
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 663683		19290823	FR	19281108 <--

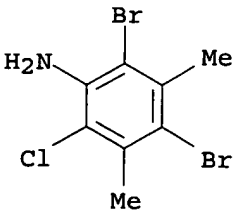
AB Azo dyes are prepared by combining the diazo compds. of halogen substitution products of 3,5-xylidine with arylamides of compds. capable of coupling and possessing affinity for vegetable fibers, such as arylamides of 3-hydroxy-2-naphthoic acid, or the β-ketocarboxylic acids. Several examples and a table of components with the colors obtained are given. The following new derivs. of 3,5-xylidine are given: 4-chloro-, m. 58-59°, 4-bromo-, m. 66-67°, 2-bromo-, m. 27-28°, b. 261-263°, 2,4-dichloro-, m. 71-72°, 2,6-dichloro-, m. 88-90°, 2,4,6-trichloro-, m. 188-189°, 4-bromo-2-chloro-, m. 70-72°, 4-chloro-2-bromo-, m. 79-80°, 2, 4-dibromo-, m. 81-82°, 2,4-dibromo-6-chloro-, m. 184-186°.

IT **854854-81-2**, 3,5-Xylidine, 2,4,6-trichloro- **854854-86-7**, 3,5-Xylidine, 2,4-dibromo-6-chloro-
(preparation of)

RN 854854-81-2 CAPLUS
CN 3,5-Xylidine, 2,4,6-trichloro- (3CI) (CA INDEX NAME)



RN 854854-86-7 CAPLUS
CN 3,5-Xylidine, 2,4-dibromo-6-chloro- (3CI) (CA INDEX NAME)

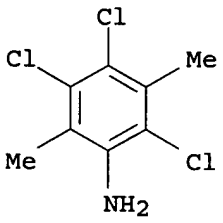


L11 ANSWER 21 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1929:31223 CAPLUS
DOCUMENT NUMBER: 23:31223
ORIGINAL REFERENCE NO.: 23:3674g-i
TITLE: 1,4-Dimethyl-3,5,6-trichloro-2-aminobenzene and some of its derivatives
AUTHOR(S): Bures, E.; Rubes, T.
SOURCE: Casopis Ceskoslovenskeho Lekarnictva (**1928**), 8, 225-31,258-64
CODEN: CCLEA3
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

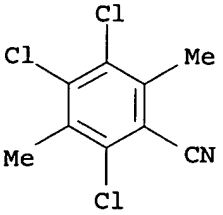
AB By the action of Cl2 on 2,5-Me2C6H3NHAc in glacial AcOH at ordinary temperature and pressure, without catalyst. B. and R. obtained 1,4-dimethyl-3,5,6-trichloro-2-acetamidobenzene m. 222°, which on hydrolysis gave the 2-amino compound, m. 206° (I). From I were prepared the Bz derivative, m. 223°, the picrate, m. above 100° (decomposition), and the 2-methylamino compound, m. 62°. There were also prepared 1,4-dimethyl-1-3,5,6-trichloro-2-hydroxybenzene, m. 175° (Me ether, m. 91°; Et ether, m. 79°; benzoate, m. 101°; acetate, m. 103%) 1,4-dimethyl-3,5,6-trichlorobenzene, m. 96°; 1,4-dimethyl-2,3,5,6-tetrachlorobenzene, m. 223°; 1,4-dimethyl-3,5,6-trichloro-2-benzonitrile, m. 213°.

IT **857974-28-8**, 2,5-Xylidine, 3,4,6-trichloro- **859202-54-3**, 2,5-Xylonitrile, 3,4,6-trichloro- **872275-60-0**, 2,5-Xylidine, 3,4,6-trichloro-N-methyl- **873979-22-7**, 2,5-Acetoxylide, 3,4,6-trichloro- **873987-55-4**, 2,5-Benzoxylide, 3',4',6'-trichloro- **876486-92-9**, Phenetole, 2,4,5-trichloro-3,6-dimethyl-
(preparation of)

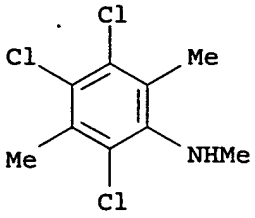
RN 857974-28-8 CAPLUS
CN 2,5-Xylidine, 3,4,6-trichloro- (5CI) (CA INDEX NAME)



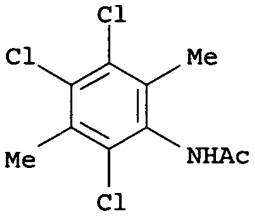
RN 859202-54-3 CAPLUS
CN 2,5-Xylonitrile, 3,4,6-trichloro- (3CI) (CA INDEX NAME)



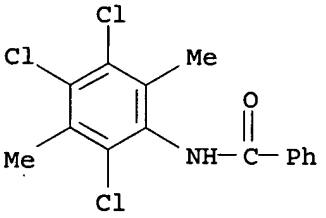
RN 872275-60-0 CAPLUS
CN 2,5-Xylidine, 3,4,6-trichloro-N-methyl- (3CI) (CA INDEX NAME)



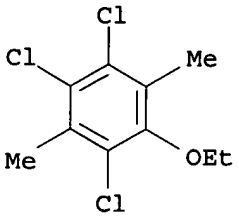
RN 873979-22-7 CAPLUS
CN 2,5-Acetoxylide, 3,4,6-trichloro- (3CI) (CA INDEX NAME)



RN 873987-55-4 CAPLUS
CN 2,5-Benzoxylide, 3',4',6'-trichloro- (3CI) (CA INDEX NAME)



RN 876486-92-9 CAPLUS
CN INDEX NAME NOT YET ASSIGNED

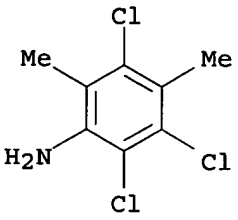


L11 ANSWER 22 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1928:37668 CAPLUS
DOCUMENT NUMBER: 22:37668
ORIGINAL REFERENCE NO.: 22:4503h-i,4504a
TITLE: 2,5,6-Trichloro-1,3-dimethyl-4-aminobenzene and some of its derivatives
AUTHOR(S): Bures, E.; Borgmann, J.
CORPORATE SOURCE: Charles Univ., Prague
SOURCE: Casopis Ceskoslovenskeho Lekarnictva (**1927**), 7, 270-80
CODEN: CCLEA3
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB By the action of Cl on 2,4-Me2C6H3NHAc, m. 123°, dissolved in glacial AcOH, at ordinary temperature and pressure, without catalysts, is formed 2,5,6-trichloro-1,3-dimethyl-4-acetamidobenzene, m. 208.5° (I).

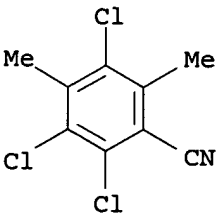
The introduction of the 3 Cl atoms stabilizes the mol. and lowers the basicity of the amine. I on saponification yields 2,5,6-trichloro-1,3-dimethyl-4-aminobenzene, m. 204° (II) (Bz derivative, m. 174-5°; HCl salt, m. 217°). II was converted to 2,5,6-trichloro-1,3-dimethyl-4-hydroxybenzene, m. 174° (Me ether, m. 91.5°; Et ether, m. 53.5°; Ac ester, m. 86°). Other derivs. of II prepared were 2,5,6-trichloro-1,3-dimethylbenzene, m. 179.5°; 2,5,6-trichloro-1,3-dimethyl-4-benzonitrile, m. 218°, which on hydrolysis gave 3,5,6-trichloro-2,4-dimethylbenzoic acid, m. 191.5°; and 2,4,5,6-tetrachloro-1,3-dimethylbenzene, m. 219°.

IT **854824-50-3**, 2,4-Xylidine, 3,5,6-trichloro- **854854-59-4**, 2,4-Xylonitrile, 3,5,6-trichloro- **854857-20-8**, 2,4-Xylidine, 3,5,6-trichloro-, -HCl **854857-71-9**, 2,4-Xylic acid, 3,5,6-trichloro- **854858-61-0**, 2,4-Xylenol, 3,5,6-trichloro-, acetate **860567-17-5**, 2,4-Benzoxylide, 3',5',6'-trichloro- **860580-56-9**, 2,4-Acetoxylide, 3,5,6-trichloro-
(preparation of)

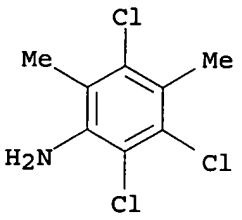
RN 854824-50-3 CAPLUS
CN 2,4-Xylidine, 3,5,6-trichloro- (3CI) (CA INDEX NAME)



RN 854854-59-4 CAPLUS
CN 2,4-Xylonitrile, 3,5,6-trichloro- (3CI) (CA INDEX NAME)

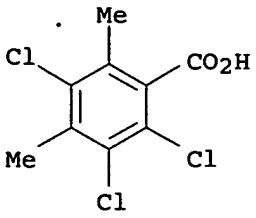


RN 854857-20-8 CAPLUS
CN 2,4-Xylidine, 3,5,6-trichloro-, -HCl (3CI) (CA INDEX NAME)

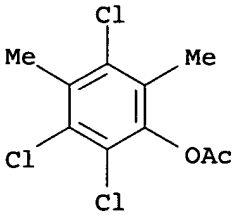


● HCl

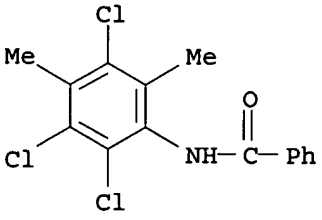
RN 854857-71-9 CAPLUS
CN 2,4-Xylic acid, 3,5,6-trichloro- (3CI) (CA INDEX NAME)



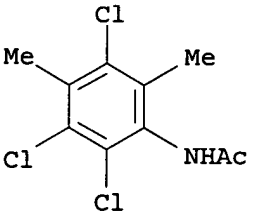
RN 854858-61-0 CAPLUS
CN 2,4-Xylenol, 3,5,6-trichloro-, acetate (3CI) (CA INDEX NAME)



RN 860567-17-5 CAPLUS
CN 2,4-Benzoxylide, 3',5',6'-trichloro- (3CI) (CA INDEX NAME)



RN 860580-56-9 CAPLUS
CN 2,4-Acetoxylide, 3,5,6-trichloro- (3CI) (CA INDEX NAME)



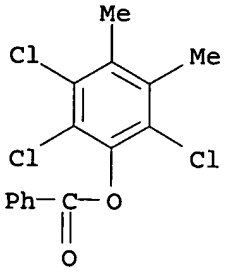
L11 ANSWER 23 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1925:1897 CAPLUS
DOCUMENT NUMBER: 19:1897
ORIGINAL REFERENCE NO.: 19:267g-i,268a
TITLE: Conversion of hydroaromatic into aromatic compounds. I. Action of chlorine on 5-chloro-1,1-dimethyl-Δ4-cyclohexen-3-one
AUTHOR(S): Hinkel, L. E.
SOURCE: Journal of the Chemical Society, Transactions (**1924**), 125, 1847-55
CODEN: JCHTA3; ISSN: 0368-1645
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB The finding that there is a divergence between the action of Cl and Br upon the same hydroaromatic compound, the former producing the more deep-seated change (C. A. 15, 56), is supported by the results of the

action of Cl on 5-chloro-1,1-dimethyl-Δ4-cyclohexen-3-one (I), which is more profound than that of Br on the corresponding Br compound (C. A. 8, 1265). Cl passed rapidly into 40 g. I in its own volume of CHCl3 until the evolution of HCl almost ceased and the liquid regained normal temperature, gave the 3,4,5-Cl3 derivative (II), m. 61°, boils with gradual decomposition, evolving HCl; alc. KOH gives 4-chloro-3,5-dihydroxy-0-xylene, m. 123° (di-Bz derivative, m. 137°); heated with H2SO4 on the H2O bath and finally at 110-20°, it gives 5,6-dichloro-0-4-xylenol, m. 102° (Bz derivative, m. 94°); heated with C9H7N for 30 mins. at 170°, there results4, 5-dichloro-0-3-xylenol, m. 95° (Bz derivative, m. 128°), which yields a tri-Cl derivative (III), m. 182.5° (Bz derivative. m. 120°) with Cl in light petroleum. II reacts with C9H7N to give 4,5-dichloro-0-3-xylenol, m. 95° (Bz derivative, m. 128°), and with Cl yields a tetra-Cl derivative, m. 127.5°. If the reaction between I and Cl is carried out in well cooled CHCl3 there results a 4,5-di-Cl derivative, b13 120-1°, having a camphoraceous odor. Action of Cl at room temperature gave II; heating gives 5,3-Cl(HO)C6H2Me2, also obtained with alc. KOH. H2SO4 gives 6-chloro-0-3-xylenol (IV), m. 98° (Bz derivative, m. 182.5°), which was synthesized from 6-chloro-4-nitro-0-xylene, slightly yellow needles, m. 101°, through 6-chloro-0-4-xylidine, m. 72°. Cl and IV in CHCl3 give III. The filtrate from II gave some II and a fraction b13 145-55°, from which a 2,4,5,6-tetra-Cl derivative of I, m. 91°, crystallized; alc. KOH gave a di-Cl xylenol, m. 119-20° (Bz derivative, m. 129°). H2SO4 yielded a mixture of tri-chloro-o-3- and 4-xylenol.

IT **861555-82-0**, 3,4-Xylenol, 2,5,6-trichloro-, benzoate (preparation of)

RN 861555-82-0 CAPLUS

CN 3,4-Xylenol, 2,5,6-trichloro-, benzoate (2CI) (CA INDEX NAME)



L11 ANSWER 24 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1921:340 CAPLUS

DOCUMENT NUMBER: 15:340

ORIGINAL REFERENCE NO.: 15:56e-i

TITLE: Action of chlorine on 3,5-dichloro-1,1-dimethyl-Δ2,4-cyclohexadiene

AUTHOR(S): Hinkel, Leonard E.

SOURCE: Journal of the Chemical Society, Transactions (**1920**), 117, 1296-1303
CODEN: JCHTA3; ISSN: 0368-1645

DOCUMENT TYPE: Journal

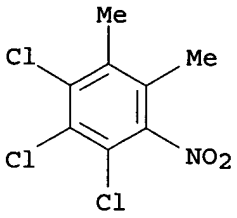
LANGUAGE: Unavailable

AB The action of Br (1 and 2 mols.) on 3,5-dichloro-1,1-dimethyl-Δ2,4-cyclohexadiene (A) has been investigated by Crossley (J. Chemical Society 85, 264(1904)) and the action of Cl on A was studied to throw more light on the mechanism of the reaction. When A in dry CHCl3 is treated with Cl, HCl is evolved and after removal of the solvent the yellow product deposited in vacuo over NaOH in colorless prisms, m. 103.5°, is 2,3,3,4,5-pentachloro-1,1-dimethyl-Δ5-cyclohexene (B), soluble in Et2O, CHCl3, Me2CO, C6H6. When B is heated at 120-30° HCl is evolved and on cooling crystals of 1,2,3,4,5-C6HMe2Cl3 (C) separated (Ber. 18, 1369(1885)). B interacted vigorously with fuming HNO3 and the product treated with H2O, washed and crystallized from AcOEt, m. 223-4°, and is 3,4,5,6-Cl4C6Me2 (D). The mother liquors from which B separated gave off HCl

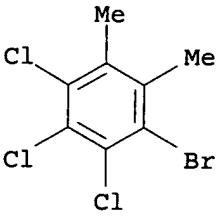
when heated to 180-200°, and when distilled gave 3,5,6-trichloro-o-xylene (E), m. 47.5°, and also C and D. When C was treated in dry CHCl3 with Fe and Cl, D resulted and with Br gave needles, m. 226°, of 3,4,5-trichloro-6-bromo-o-xylene (F), readily soluble in Et2O, C6H6, and sparingly soluble in CHCl3 and alc. When C is treated with fuming HNO3 and the product is washed with H2O and crystallized from alc. stout yellowish needles, m. 149°, of 3,4,5-trichloro-6-nitro-o-xylene (G) are formed. This product is soluble in Et2O, C6H6, CHCl3 and light petroleum. C was prepared from o-4-xylidene (Crossley, loc. cit.) by acetylating, chlorinating and treating in HCl with CuCl and NaNO2. When A in cold CHCl3 was treated with Cl, no crystalline product could be secured and on distilling the liquid, 3 fractions (1) 217-20°, (2) 222-6°, and (3) 228-33°, were obtained. Fraction 2 gave a product which on treatment with Br and Fe gave 3,5,4,6-Cl2Br2C6Me2 (H) (loc. cit.) and fraction 3 contained E. When E was treated in CHCl3 with Br and Fe, H was formed, and when treated with fuming HNO3 the resulting product was 3,5,4,6-Cl2(O2N)2C6Me2.

IT **861555-92-2**, o-Xylene, 3,4,5-trichloro-6-nitro-
861614-30-4, o-Xylene, 3-bromo-4,5,6-trichloro-
(preparation of)

RN 861555-92-2 CAPLUS
CN o-Xylene, 3,4,5-trichloro-6-nitro- (2CI) (CA INDEX NAME)



RN 861614-30-4 CAPLUS
CN o-Xylene, 3-bromo-4,5,6-trichloro- (2CI) (CA INDEX NAME)



L11 ANSWER 25 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1912:5489 CAPLUS
DOCUMENT NUMBER: 6:5489
ORIGINAL REFERENCE NO.: 6:859f-i,860a
TITLE: s-Iodopseudocumene and Derivatives
AUTHOR(S): Willgerodt, C.; Meyer, R.
CORPORATE SOURCE: Univ. Freiburg
SOURCE: Ann. (**1912**), 385, 341-51
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

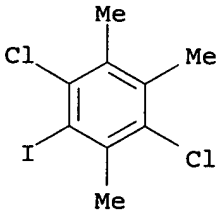
AB s-Iodopseudocumene, prepared by dissolving 11 g. pseudocumene in 100 cc. petroleum ether and b. with 20 g. sulfur iodide in 100 cc. HNO3 (1.34) 4 hrs. on the water bath. M. 37°. s-Pseudocumyliodide chloride, C9H11Cl2I, yellow crystals, decompose, 66°. s-Iodosopseudocumene, C9H11OI, yellowish white powder, decompose, 171°. Acetate, needles, m. 123°. s-Iodopseudocumene, C9H11O3I, jelly-like, explodes about 210°. Di-s-pseudocumyliodinium; compounds. Hydroxide, could not be obtained pure. Hydrochloride, crystalline powder, m. 107°. Chloroplatinate, crystalline powder, m. 159°. Chloroaurate, needles from alc., m. 90°. Hydrobromide needles from alc., m. 118°.

Hydriodide, m. 120°. Dichromate, amorphous precipitate, explodes at 120°. Monoiado-di-s-pseudocumyliodinium compds. Anion = C9H(C9H10I. Hydrochloride, m. 106°. Chloroplatinate, m. 150°. Chloromercurate, amorphous, m. 108°. Chloroaurate, amorphous, Hydrobromide, bright yellow amorphous mass, m. 105°. Hydriodide, amorphous powder, m. 112°. Dichromate, decamp. 113°. Phenyl-s-pseudocumyliodinium compds. Anion = PhC9H11I. Hydrochloride, crystalline powder, m. 186°. Chloroplatinate, amorphous, m. 188°. Chloroaurate, crystalline precipitate, m. 117°. Chloromercurate, needles from alc., m. 161°. Hydrobromide, needles from H2O, m. 173°. Hydriodide, crystalline, decompose 147°. Dichromate, crystalline powder, explodes at 184°. p-Tolyl-s-pseudocumyliodinium compds. Anion = C7H7(C9H11)I. Hydrochloride, needles from H2O, m. 171°. Chloroplatinate, leaves from alc., decompose about 165°. Chloroaurate, needles, m. 71°. Chloromercurate, needles, m. 81°. Hydrobromide, crystalline powder, m. 148°. Hydriodide, decompose 108°. Dichromate, needles, decompose 149°. Dichlorobinyl-s-pseudocumyliodinium compds. Anion = CHCl = CCl(C9H11)I. Hydrochloride, m. 169°. Chloroplatinate, crystalline powder, decompose 150°. Chloroaurate, crystalline, m. about 134°. Hydrobromide, amorphous, m. 131°. Hydriodide, amorphous, m. 96°. p-Dichloro-s-iodopseudocumene, C9H9Cl2I, beautiful crystals from C6H6 m. 188-9°. Attempts to prepare the iodide chloride failed.

IT **859954-77-1**, Pseudocumene, 3,6-dichloro-5-iodo-
(preparation of)

RN 859954-77-1 CAPLUS

CN Pseudocumene, 3,6-dichloro-5-iodo- (1CI) (CA INDEX NAME)



L11 ANSWER 26 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1910:4457 CAPLUS

DOCUMENT NUMBER: 4:4457

ORIGINAL REFERENCE NO.: 4:752d-h

TITLE: Acetylization with Acetic Anhydride and Sulphuric Acid

AUTHOR(S): Blanksma, J. J.

SOURCE: Amsterdam. Chem. Weekblad (**1910**), 6, 717-27

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

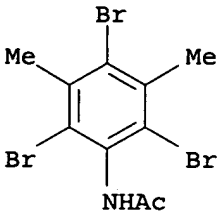
AB By addition of a drop of concentrate H2SO4 to the reaction mixture, the acetylization of PhNH2 and its OH derivative is greatly accelerated, the following Ac2 derivs. being thus prepared, some, within a few min.: Di-acetyl-o-nitroaniline, colorless crystals, m. 94°. 2,4,6-Trinitro-m-diacetophenylene-diamine, colorless crystals, decompose 300°. 2,4,6-Tribromo-m-acetotoluide, colorless crystals, m. 205°, and the diacetyl derivative, colorless crystals, m. 103°. The former, with HNO3, yields 2,4,6,tribromo-5-nitro-m-acettoluide, colorless needles, m. 261°, which, heated with concentrate H2SO4, gives 2,4,6-tribromo-5-nitro-m-toluidine, light yellow needles, m. 184°. Diacetyl derivative, colorless crystals, m. 188°. 2,4,6-Tribromo-sym-acetoxylidide, colorless crystals, m. 258°. 2,4,6-Tribromo-3,5-acetonitroaniline, colorless needles, m. 275°, which, by boiling with Ac2O and H2SO4, gives the diacetyl derivative, m. 165°. From 4-nitro-o-toluidine, with AcOH and Br., 3,5-dibromo-4-nitro-o-toluidine, yellow crystals, m. 104°. Acetyl derivatives, colorless crystals, m. 201°. Diacetyl derivative, m. 159°. The monoacetyl derivative, with HNO3 and H2SO4, gives 4,6-dinitro-3,5-dibromoacetotoluide, m. 280°. Similarly,

3,5-dibromo-2-nitro-ptoluidine, yellow crystals, m. 82°, and 2,6-dinitro-3,5-dibromo-p-acetotoluide, colorless crystals, m. 275° (KUNCKELL, 267°), which, with concentrate H_2SO_4, gives, 2,4-dinitro-3,5-dibromo-p-toluidine, dark yellow needles, m. 174°. Of the substituted phenol derivs. were prepared 2,4-dinitrophenyl acetate, colorless crystals, m. 72°, and 2,4-dibromo-6-nitrophenyl acetate, colorless crystals, m. 88°. Similarly, δ-methylfurfurylidene diacetate, large colorless, transparent crystals, m. 25°, and triacetyl β-hydroxy-δ-methylfurfuraldehyde, colorless, transparent crystals, from ligroin, m. 73°.

IT **861611-09-8**, 3,5-Acetoxylide, 2,4,6-tribromo-
(preparation of)

RN 861611-09-8 CAPLUS

CN 3,5-Acetoxylide, 2,4,6-tribromo- (1CI) (CA INDEX NAME)



L11 ANSWER 27 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1908:13564 CAPLUS

DOCUMENT NUMBER: 2:13564

ORIGINAL REFERENCE NO.: 2:2952g-i,2953a-b

TITLE: Preparation of Halogen-Substituted Anilides

AUTHOR(S): Mannino, A.; Didonato, L.

CORPORATE SOURCE: Ist. chim. R. Univ. Roma

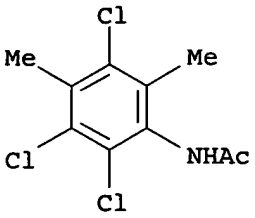
SOURCE: Gazzetta Chimica Italiana (**1908**), 38(2), 20-31
CODEN: GCITA9; ISSN: 0016-5603

DOCUMENT TYPE: Journal

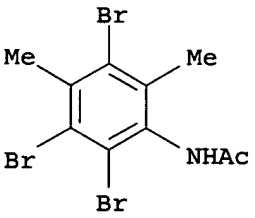
LANGUAGE: Unavailable

AB A systematic study of the action of mixtures of concentrate HCl + HNO_3 and of HBr + HNO_3 upon acetanilide and its homologues confirms and extends the observations of Schloss (Diss. Lausanne 1901) and of Verda (Gazz. chim. ital., 32, [2], 20). In each case a single halo-gen-substituted amide is produced, without by-products and without the elimination of acetyl. Acetanilide with HCl and HNO_3 produces, on heating, 2,4-dichloracetanilide, but, if the reaction is carried on farther, all is converted into 2,4,6-trichloracetanilide; the latter is also obtained starting with o- or p-chloracetanilide, while m-chloracetanilide yields 3,5-dichloracetanilide. The HBr mixture produces with the same substances, in the above order, 2,4-dibromacetanilide, 2-chlor-4-bromacetanilide, 4-chlor-2-bromacetanilide, and a 3-chlor-2(?)-bromacetanilide, m. 105-7°. From m-bromacetanilide with the HCl mixture there result brilliant flesh-colored needles, m. 194-5°, which are probably 4,6-dichlor-3-bromacetanilide; with the HBr mixture, 2,4,5-tribromacetanilide, white needles, m. 188-9°. Paranitracetanilide yields 2-chlor-4-nitro-acetanilide, and an unidentified bromine compound. Acetyl-o-toluidine yields dichloracetyl-o-toluidine and 5-brom-acetyl-2-toluidine as shown by Verda, but the p-compound takes up 3 Cl or 2 Br. Acetyl-m-xylidine yields trichloracetylmetaxylidine, white needles, m. 190-2°, and tribromacetylmetaxylidine, m. 246-8°. Acetyl-α-naphthylamine, with aqua regia takes up 1 Cl and 1 NO, as already observed by Verda. Saponification with 15% HCl produces a chloronitro-α-naphthylamine, yellow needles, m. 230°; with the HBr mixture, 3,8-dibrom-acetyl-α-naphthylamine is formed. Acetyl-β-naphthylamine yields a tribrominated substance, m. 250°, as also does 1,3,6-tribromacetyl-β-naphthylamine; but, on saponification, a base is formed, m. 125°, while the 1,3,6-tribrom-β-naphthylamine m. 143°, according to Claus and Philipson (J. pr.

Chemical [2], 43, 56).
IT * **860580-56-9**, 2,4-Acetoxylide, 3,5,6-trichloro- **861611-23-6**
, 2,4-Acetoxylide, 3,5,6-tribromo-
(preparation of)
RN 860580-56-9 CAPLUS
CN 2,4-Acetoxylide, 3,5,6-trichloro- (3CI) (CA INDEX NAME)

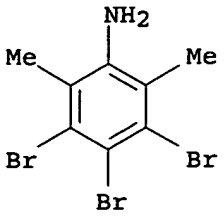


RN 861611-23-6 CAPLUS
CN 2,4-Acetoxylide, 3,5,6-tribromo- (1CI) (CA INDEX NAME)

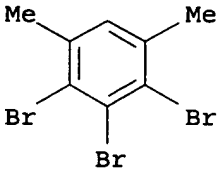


L11 ANSWER 28 OF 28 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1907:1262 CAPLUS
DOCUMENT NUMBER: 1:1262
ORIGINAL REFERENCE NO.: 1:328i,329a-c
TITLE: The Three Isomeric Tribromxylenes
AUTHOR(S): Jaeger, F. M.; Blanksma, J. J.
CORPORATE SOURCE: Univ. of Amsterdam
SOURCE: Recueil des Travaux Chimiques des Pays-Bas et de la Belgique (**1907**), 25, 352-63
CODEN: RTCPB4; ISSN: 0370-7539
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB A. Tribrom-o-xylenes. Bromination of the xylidines 1,2,3 and 1,2,4 gave 4,6-dibrom-1,2,3-xylidine, m. 56°, and 3,5-dibrom-1,2,4-xylidine, m. 63°. Replacing the amino group in each by bromine yielded 3,4,6-tribrom-o-xylene, m. 86, and 3,4,5-tribrom-o-xylene, m. 105°. Bromination of 1,2,4-acetxylidide gave 3,6-dibrom-1,2,4-xylidine, m. 65°, which in turn gave the 3,4,6-tribrom-o-xylene. B. Tribrom-m-xylenes. The 2,4,6-tribrom-m-xylene, m. 85°, was obtained from 1,3,5-xylidine, 4,6-dibrom-2-aminoxylene 2-aminoxylene and 1,3,4-acetxylidide. 2,4,6-Tribromxylidine, m. 195°. 6-Brom-4-amino-m-xylene, m. 96°. 2-6-Dibrom-4-aminoxylene, m. 65°. 4,5,6-Tribrom-m-xylene, m. 105°, came from 4,6-dibrom-2-amino-m-xylene and 6-brom-4-amino-m-xylene. 4,5,6-Dibrom-2-amino-m-xylene, m. 197°. 4,6-Dibrom-m-xylene, m. 69°. 4,6-Dibrom-2,5-dinitro-m-xylene, m. 252°. 4,5-Dibrom-m-xylene, b. 256°, m. 11°. 4,5-Dibrom-2,6-dinitroxylene, m. 193°. 2,6-Dibrom-4-amino-m-xylene, m. 65°. 5,6-Dibrom-4-amino-m-xylene, m. 38°, 2,4,5-Tribrom-m-xylene, m. 87°, was obtained from 4-brom-2-amino-m-xylene. 4,5-Dibrom-2-amino-m-xylene, m. 51°. C. Tribrom-p-xylene, m. 89°, was obtained by bromination of 1,4,5-xylidine, followed by replacement of the amino group by bromine.
IT **860747-80-4**, 2,6-Xylidine, 3,4,5-tribromo- **860748-31-8**, m-Xylene, 4,5,6-tribromo- **860748-32-9**, m-Xylene, 2,4,5-tribromo-
(preparation of)

RN 860747-80-4 CAPLUS
CN 2,6-Xylidine, 3,4,5-tribromo- (1CI) (CA INDEX NAME)



RN 860748-31-8 CAPLUS
CN m-Xylene, 4,5,6-tribromo- (1CI) (CA INDEX NAME)



RN 860748-32-9 CAPLUS
CN m-Xylene, 2,4,5-tribromo- (1CI) (CA INDEX NAME)